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THE CHEMICAL EXAMINATION OF WATER, SEWAGE AND FOODS

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CAMBRIDGE PUBLIC HEALTH SERIES

UNDER THE EDITORSHIP OF

G. S. Graham-Smith, M.D. and J. E. Purvis, M.A.

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University Lecturer in Chemistry and Physics in their application to Hygiene and Preventive Medicine, and Secretary to the State Medicine Syndicate

THE CHEMICAL EXAMINATION OF WATER, SEWAGE, FOODS AND OTHER SUBSTANCES

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THE CHEMICAL EXAMINATION OF WATER, SEWAGE, FOODS AND OTHER SUBSTANCES

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EDITORS' PREFACE

IN view of the increasing importance of the study of public hygiene and the recognition by doctors, teachers, administrators and members of Public Health and Hygiene Committees alike that the *salus populi* must rest, in part at least, upon a scientific basis, the Syndics of the Cambridge University Press have decided to publish a series of volumes dealing with the various subjects connected with Public Health.

The books included in the Series present in a useful and handy form the knowledge now available in many branches of the subject. They are written by experts, and the authors are occupied, or have been occupied, either in investigations connected with the various themes or in their application and administration. They include the latest scientific and practical information offered in a manner which is not too technical. The bibliographies contain references to the literature of each subject which will ensure their utility to the specialist.

It has been the desire of the editors to arrange that the books should appeal to various classes of readers : and it is hoped that they will be useful to the medical profession at home and abroad, to bacteriologists and laboratory students, to municipal engineers and architects, to medical officers of health and sanitary inspectors and to teachers and administrators.

Many of the volumes will contain material which will be suggestive and instructive to members of Public Health and Hygiene Committees ; and it is intended that they shall seek to influence the large body of educated and intelligent public opinion interested in the problems of public health.

AUTHORS' PREFACE

THIS book is intended for the use of students who are attending courses of instruction for diplomas and degrees in Public Health, and for those who are studying the chemistry of water, sewage, sewage effluents, foods, disinfectants, etc. during their ordinary laboratory practice. It will probably be also useful to those who are engaged as public analysts or in public health matters generally.

It does not give an exhaustive account of all the available methods of examination, but it describes those which have been used and tested by the authors both in laboratory instruction and in the daily work of a public analyst. Full references are given in the text to the sources of the different methods, and, at the end, there is a short bibliography of some of the more important works which discuss the various subjects. No illustrations have been introduced, as the apparatus, as well as the appearance and characters of the substances which require the use of a microscope, can only be fully understood and appreciated in the actual practical work of the laboratory.

A considerable number of typical analyses have been introduced to illustrate the variations which may occur in the composition of the articles themselves. We desire to convey our grateful thanks to those who have allowed us to insert the results of their own work and which we have acknowledged by name in the appropriate places.

The methods employed in the bacteriological examination of water, sewage, foods, air, etc. is the subject of a separate volume in this series by Dr W. G. Savage.

J. E. P.
T. R. H.

CAMBRIDGE,
July, 1914.

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CHAPTER I

WATER—SEWAGE—SEWAGE EFFLUENTS—RIVER-WATER AND SEWAGE—SEA-WATER AND SEWAGE

WATER.

Chemically pure water does not occur in nature. All water supplies are obtained ultimately from rain water, which contains various substances, both organic and inorganic, dissolved by the water during its passage through the air, soil, subsoil, etc.; and, it may be, by contamination with sewage material and trade wastes.

An analysis of water is undertaken with the object of estimating these impurities and of deciding whether the water is of sufficient purity to be used for domestic purposes. The value of this analysis depends on the accuracy of the results, and, in a large measure, on careful and proper sampling.

Method of sampling. The sample should be collected in a "Winchester Quart," having a ground glass stopper. Stone jars should not be used, and care should be taken to see that the bottle has not been previously used for storing ammonia and ammonium salts. Before collecting the sample, the bottle should be well rinsed at least three times with the water.

If the sample is to be taken from a stream, the bottle should be completely immersed in the stream as near the centre as possible, and care should be taken not to disturb any mud.

If the sample is to be taken from a well, at least ten gallons should be allowed to run to waste before filling the bottle, and the water should then be allowed to flow directly into the bottle; in no case should a funnel be used.

When the sample is to be collected from a tap, at least

ten gallons should be allowed to run to waste ; and the sample should then be collected directly from the tap.

Physical characters. A careful preliminary examination of a water is of great importance.

(*a*) Test the water with blue and red litmus papers, and notice if the reaction is acid or alkaline. Most drinking waters are slightly alkaline or neutral in reaction. Peaty waters and waters from upland surfaces are often acid, as well as waters from chemical works and breweries. Waters which are contaminated with sewage are usually alkaline. Waters which contain carbonic dioxide give a slightly acid reaction.

(*b*) Warm a portion in a test-tube and notice if any smell is produced. Polluted water may give off sulphuretted hydrogen.

(*c*) Place a portion in a two-foot glass tube and examine it downwards in a good light. Notice if it is clear and bright or turbid. Pure water has a bluish tinge ; polluted waters may be yellow or brown ; peaty waters and upland surface are usually coloured yellow or brown.

(*d*) Allow a portion of the water to stand for an hour or two in a conical shaped tube, and collect the sediment. Mount a little of the sediment on a glass slide, and examine it carefully with a microscope to see if it is mineral matter, such as sand or chalk or oxide of iron ; or vegetable matter, such as vegetable cells, starch granules, cotton or linen or silk fibre, yeasts, moulds, bacteria, diatoms and other water organisms, or animal refuse such as worms, insects, hairs, fish scales, animal fibre, etc.

(*e*) Also notice if it is well ærated, by shaking a portion of it in a glass bottle and noticing the liberation of gas bubbles.

Free and saline ammonia.

Solutions required :

Nessler's solution. Weigh out

35 grams of potassium iodide,
13 grams of mercuric chloride,
120 grams of caustic potash,

dissolve each separately in water, and gradually add the potassium iodide solution to the solution of mercuric chloride, a precipitate of mercuric iodide appears at first which is afterwards redissolved in the excess of potassium iodide ; the caustic potash solution is then added gradually ; and, afterwards, a saturated solution of mercuric chloride drop by drop, until a very slight precipitate, which will not redissolve, is formed. The whole is then finally made up to 1000 c.c. and allowed to stand until the precipitate settles ; the solution should always be kept in contact with the precipitate.

Ammonium chloride solution (strong) : weigh out 3·147 grams of pure dry ammonium chloride, dissolve it in water and make up to 1000 c.c.

Ammonium chloride solution (dilute) : measure out 100 c.c. of the strong ammonium chloride solution and add distilled water to 1000 c.c. 1 c.c. of this solution = 0·0001 gram of ammonia.

Estimation of free and saline ammonia. The most convenient apparatus for this determination is a 1500 c.c. round bottomed distilling flask, with a ground glass stopper, having a fused-in delivery tube, bent at right angles, this delivery tube is fitted into a double surface condenser.

It is necessary first to free the apparatus from ammonia ; and, in order to do this, 200 c.c. of distilled water and about one gram of solid sodium carbonate, which has been fused in a platinum dish to drive off any ammonium salts, are placed in the distilling flask and boiled ; the distillate is collected in a Nessler cylinder, and the boiling continued until the distillate gives no colour with 2 c.c. of the Nessler solution. As soon as the apparatus has been freed from ammonia, 500 c.c. of the sample are added to the contents of the distilling flask and the boiling continued ; the distillate being collected in Nessler cylinders, 50 c.c. at a time ; to the contents of each cylinder, 2 c.c. of the Nessler solution are added and the colour obtained is compared with the standard colours.

Standard colours are obtained by placing known quantities of the dilute ammonium chloride solution (*e.g.* 0·5 c.c., 1·0 c.c., 1·5 c.c., etc.) in a Nessler cylinder, filling up to the mark with

distilled water free from ammonia, and adding 2 c.c. of the Nessler solution. In some cases it may be found that the colour obtained with the first glass of the distillate is too deep to be compared with a standard solution, the contents of the glass are therefore diluted to 250 c.c. and then compared with the standard ; or an aliquot portion may be taken and diluted with ammonia free water to 50 c.c. and then compared with the standard. The amount of ammonia obtained in the distillation is calculated from the known strength of the dilute ammonia solution.

In this process the sodium carbonate decomposes any ammonium salts in the water, and the ammonia so obtained, together with any free ammonia in the water, passes over and is collected in the distillate. Ammonia is one of the first products of the decomposition of animal matter and its presence in water in very large quantities must be looked upon with suspicion ; but it must be remembered that ammonium salts are not in themselves harmful and their presence in water does not necessarily render the sample unfit for drinking purposes.

Albuminoid ammonia.

Solution required :

Alkaline permanganate solution. Weigh out

8 grams of potassium permanganate,
200 grams of caustic potash,

dissolve these together in about 1200 c.c. of distilled water and evaporate the solution to about 900 c.c., add 200 c.c. of distilled water and again evaporate the solution to 1000 c.c., the solution, which will be found to be free from ammonia, is kept in a glass stoppered bottle.

Estimation of albuminoid ammonia. To the remainder of the water in the distilling flask, after the estimation of free and saline ammonia, add 100 c.c. of the alkaline permanganate solution which has been previously heated to boiling and continue the boiling ; the ammonia is estimated colourimetrically as before.

The nitrogen in organic combination (as urea, uric acid, indol, skatol, and other proteid substances) is oxidised by the action of the alkaline solution of permanganate to ammonia. The amount of ammonia obtained is, therefore, a measure of the organically combined nitrogen present in the sample.

Estimation of total solid matter.

Measure out 100 c.c. of the sample into a weighed platinum dish, and evaporate it to dryness on a water bath; then place the dish in a water oven at 100° C. for three hours; cool in a desiccator and weigh; the increase in weight represents the total solid matter in 100 c.c. of the water. Each mgm (=0·001 of a gram) of solid matter represents one part per 100,000.

Chlorine as chlorides.

Solutions required:

Silver nitrate solution. Weigh out 2·4 grams of pure dry silver nitrate, and dissolve it in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 0·0005 gram of chlorine; or when working on 50 c.c. 1 c.c. = 1 part of chlorine per 100,000.

Potassium chromate solution. Weigh out 10 grams of potassium chromate, free from chloride, and dissolve it in 100 c.c. of distilled water.

Estimation of chlorine as chlorides. Place 50 c.c. of water in a porcelain dish; add three drops of the potassium chromate solution, and run in the silver nitrate solution from a burette, with constant stirring, until a slight but permanent red brown colour is obtained; then read off the number of c.c. of silver nitrate solution used. The silver nitrate precipitates the chlorine present in the water, as silver chloride; and, as soon as all the chloride is precipitated, a reddish insoluble compound of silver chromate is formed.

Oxygen absorbed from potassium permanganate solution.

(1) *Solutions required:*

Potassium permanganate solution. Weigh out 0·395 gram of pure potassium permanganate; dissolve it in distilled water

and make up to 1000 c.c. 1 c.c. of this solution = 0·0001 gram of available oxygen.

Oxalic acid solution. Weigh out 0·7875 gram of crystallized oxalic acid; dissolve it in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 1 c.c. of the permanganate solution.

Sulphuric acid solution. Measure out 33 c.c. of pure concentrated sulphuric acid, and add it gradually, with constant stirring, to 66 c.c. of distilled water; allow it to cool; to the cold solution add, drop by drop, potassium permanganate solution until a faint pink colour remains after keeping the solution at 80° F. for four hours.

Estimation of the oxygen absorbed. Into each of two conical flasks, of about 800 c.c. capacity, pour a small quantity of concentrated sulphuric acid and thoroughly rinse, taking care that the sulphuric acid comes in contact with the whole of the inside of the flask; wash out the flasks thoroughly first with tap water and afterwards with distilled water.

Into one flask measure out 250 c.c. of the sample; into the other flask 250 c.c. of distilled water; and to each add 10 c.c. of the potassium permanganate solution from a pipette, and 10 c.c. of the sulphuric acid solution; raise the temperature to 80° F. and maintain it at this temperature for four hours; if, at any time, the pink colour disappears a further quantity of 10 c.c. of the potassium permanganate solution must be added. At the end of four hours titrate the solution in each flask with the oxalic acid solution; the difference in the two titrations multiplied by 0·0001 will give the amount of oxygen absorbed by 250 c.c. of the sample.

(2) *Potassium iodide and sodium thiosulphate method.*

Solutions required:

Standard solution of potassium permanganate. } prepared as described above.
Dilute sulphuric acid solution. }

Potassium iodide solution; 10 grams of KI are dissolved in 1000 c.c. of water.

Standard sodium thiosulphate solution; 1 gram of pure

re-crystallized sodium thiosulphate is dissolved in 1000 c.c. of water.

Starch solution; 1 gram of pure potato starch is mixed into a smooth paste with a small quantity of water; and 200 c.c. of boiling water are then carefully added with constant stirring and the solution allowed to cool.

The process. The actual determination is carried out exactly as described above, except that after standing for four hours at 80° F. a few drops of the potassium iodide solution are added to each flask, when a brown solution will be obtained owing to the reaction of the potassium iodide with the excess of potassium permanganate and the liberation of free iodine; the standard solution of sodium thiosulphate is then run in from a burette with constant shaking until a straw yellow colour is obtained; a few drops of starch solution are then added, producing a deep blue colour, and the titration with the thiosulphate solution continued until the blue colour is discharged.

Example. A blank experiment required 34 c.c. of the thiosulphate solution, and 250 c.c. of a water required 31·3 c.c.

$400 \times 0{\cdot}001 \times \frac{34-31{\cdot}3}{34} = 0{\cdot}032$ parts of oxygen absorbed per 100,000 parts of water.

The purer natural waters, as a rule, contain only very small quantities of oxidizable organic substances of vegetable origin; this process, therefore, is a measure of the oxidizable organic substances of animal origin in the water.

With view of a coming to a provisional conclusion more rapidly than can possibly be done in all bacteriological tests, and to a certain extent with chemical tests, Dr A. C. Houston, in his 5th Report (1911) and 7th Report (1913) to the London Metropolitan Water Board, made a considerable number of detailed experiments to correlate the oxygen absorbed from potassium permanganate in *five minutes*, as compared with that absorbed in three hours, both at 80° F. He found that, taking the arbitrary figure 0·1 per 100,000 as the basis for objecting to a water in a three hours' test, the comparable figure for the five minutes' test was somewhat less than 0·038, the ratios varying slightly with different waters. When a water is appreciably

worse than 0·1 in the three hours' test, the five minutes' test will be usually above 0·038; and when a water is appreciably better than 0·1 in the three hours' test, the five minutes' test may be nearly always trusted not to exceed 0·038. Dr Houston states that since April, 1910, the figure 0·038 has been adopted for the five minutes' test, and if on the examination of a sample of water, the results reach or exceed this amount, a telephone communication is sent at once to the district concerned. The five minutes method is of little use when the waters are very good, but when they are unsatisfactory, the parallelism between the two sets of results is very striking, although, of course, the *actual* results differ in the two cases.

Nitrogen as Nitrates.

Estimation of nitrogen as nitrates. Nitrogen occurring as nitrates is determined by one of several methods:

(*a*) Gasometric method.
(*b*) Colourimetric method.
(*c*) Ammonia method.
(*d*) Indigo method.

(*a*) *Gasometric method.* The residue from the determination of the total solid matter is dissolved in the smallest possible quantity of water, the sides of the dish being well scrubbed with a rubber-tipped glass rod; the solution is then poured into the cup of a Crum's nitrometer, filled with mercury and standing in a dish containing mercury, the tap of the nitrometer is carefully opened and the solution allowed to run into the nitrometer; an equal volume of concentrated sulphuric acid is then poured into the cup and allowed to flow into the nitrometer in its turn, care being taken that no air is admitted; the bottom of the tube is closed with the thumb and the whole tube removed from the dish and well shaken, at an angle of 45° so that the mercury comes in contact with the mixture at the top of the tube; from time to time during the shaking the tube is replaced in the dish and the thumb removed to release the pressure; when the whole of the nitrate is decomposed the volume of gas obtained (nitric oxide) is noted, together with

the temperature and pressure, and the volume is reduced to normal temperature and pressure. From the volume of the gas thus obtained the weight of nitrogen as nitrate in 100 c.c. of the water is calculated. The nitrogen present as nitrate is converted by the action of the sulphuric acid and mercury into nitric oxide (NO).

(*b*) *Colourimetric method. Solutions required:*

Phenol-sulphuric acid solution; weigh out 24 grams of pure phenol; dissolve it in 12 c.c. of distilled water and add 148 c.c. of pure concentrated sulphuric acid; heat the mixture on the water bath in a round bottomed flask for six hours; allow it to cool and preserve it in a tightly stoppered bottle.

Potassium nitrate solution; weigh out 0·722 gram of pure fused potassium nitrate; dissolve it in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 0·0001 gram of nitrogen.

Place 25 c.c. of the water in a glass or porcelain dish and evaporate it to dryness on the water bath. 5 c.c. of the standard potassium nitrate solution are evaporated in another similar dish.

To each dish, when cold, 2 c.c. of phenol-sulphuric acid are added, and the contents of each dish thoroughly mixed with a glass rod; to each dish is then added 25 c.c. of distilled water, and the contents are washed with dilute ammonia solution into Nessler cylinders; excess of ammonia is added to each solution to the 50 c.c. mark. A deep yellow colour will appear in the standard solution and also in the water if nitrates are present; these colours are compared and the volume of the darker coloured solution equal in tint to 50 c.c. of the paler solution is noted. From this the amount of nitrate in 25 c.c. of the water is calculated, as 5 c.c. of the standard potassium nitrate solution containing 0·0001 gram of nitrogen were taken. When phenol-sulphuric acid acts upon a nitrate, picric acid is formed, and this with ammonia forms ammonium picrate, which has a deep yellow colour, clearly visible in even very dilute solutions.

(*c*) *Ammonia method. Preparation of copper-zinc couple.* A small piece of zinc foil is immersed in a solution of copper sulphate containing 3 per cent. of the dissolved crystallized salt; a deposit of copper is left upon the zinc; the deposition of the copper should not be allowed to proceed too far,

otherwise it will not adhere firmly to the zinc; the couple is well washed with distilled water.

250 c.c. of the sample are made acid with 0·5 gram of crystallized oxalic acid; the wet couple is added; the whole placed in a water bath of cold water and the temperature gradually raised to and maintained at 60° C. for one hour; the ammonia formed is then distilled off in the ammonia distilling apparatus and estimated colourimetrically with Nessler solution in the usual way.

The copper-zinc couple decomposes the water with the formation of hydrogen, which reacts on the nitrate to form nitrite; and this, in its turn, is further reduced by the hydrogen, forming ammonia.

Although this method is described it should be stated that it has been proved by Purvis and Courtauld[1] that the method is only reliable provided the waters contain merely inorganic substances in solution, and little or no substances of an organic nature. When sewages, or sewage effluents, or waters polluted by animal or vegetable refuse were examined for nitrates by the copper-zinc couple method, it was proved by them that the effect of the couple was to produce ammonia by the reduction of the dissolved organic substances. The oxygen liberated by the couple, as well as the hydrogen, influenced the decomposition and the degradation of the dissolved organic compounds, and produced ammonia as one of the final products, in addition to a reduction of the nitrates, whereby a higher value is given to the nitric nitrogen present in any solution. The method must, therefore, be rejected, for the ammonia thus produced comes from the dissolved nitrogenous compounds as well as from the nitrates and nitrites.

(*d*) *Indigo method* (*Maix-Trommsdorf method*).

Solutions required:

Dilute indigo solution; 1 gram of pure powdered indigo is mixed with 5 to 10 c.c. of fuming sulphuric acid in a small mortar and allowed to stand for two hours; the mixture is then diluted with 300 c.c. of water and filtered. 10 c.c. of this solution are diluted to 100 c.c. for use. This solution must be kept in the dark.

[1] *Proc. Camb. Phil. Soc.* (1908), vol. XIV, p. 441.

Concentrated sulphuric acid free from nitrates.

Potassium nitrate solution. 0·0749 gram of potassium nitrate is dissolved in 1000 c.c. of water; 25 c.c. of this solution contain 0·001 gram of N_2O_5.

To determine the strength of the indigo solution, 25 c.c. of the potassium nitrate solution are placed in a small flask together with 25 c.c. of the concentrated sulphuric acid, and the indigo solution is run in from a burette, with constant shaking, until a permanent green colour is obtained; a second experiment is then made with a further 25 c.c. of the potassium nitrate solution, but in this case the whole of the indigo solution required in the first experiment is run in at once and the mixture well shaken. Should the green colour not be permanent, further quantities of the indigo solution are added drop by drop until the permanent colour is obtained. From the second experiment the number of c.c. of indigo solution corresponding to 0·001 gram of N_2O_5 is obtained; this should be from 7 to 9 c.c.

The process for the sample of water is carried out exactly as described above, 25 c.c. of water being used instead of the potassium nitrate solution. The method is very rapid, but the results obtained are not absolutely accurate as they are affected by the presence of easily oxidizable organic matter, and also by the rapidity with which the determination is carried out.

When animal organic matter containing nitrogen is oxidized, nitrates are ultimately formed; the presence, therefore, of large quantities of nitrogen in the form of nitrates in a water indicates past pollution, unless it can be shown that such nitrates are obtained from the rocks in the well. For example, waters from the chalk contain as much as 0·7 part per 100,000 of nitric nitrogen.

Nitrogen as Nitrites.

(*a*) *Solutions required:*

Metaphenylene-diamine solution. Weigh out 2 grams of metaphenylene-diamine hydrochloride; dissolve it in distilled water and make up to 100 c.c.

Standard potassium nitrite solution. Weigh out 0·406 gram of pure silver nitrite ; dissolve it in boiling distilled water, and add excess of pure potassium chloride ; cool and make up to 1000 c.c. ; allow the precipitate of silver chloride to settle, and take 100 c.c. of the clear solution and dilute to 1000 c.c. ; one c.c. of this solution = 0·00001 gram of N_2O_3. This solution will not keep long and has to be renewed constantly.

Place 100 c.c. of the water in a Nessler cylinder ; add to it 2 c.c. of the metaphenylene-diamine solution, and 2 c.c. of 25 per cent. sulphuric acid ; allow it to stand for at least thirty minutes ; if a colour is produced, compare it with the colour obtained by treating known volumes of the standard nitrite solution in a similar manner.

Metaphenylene-diamine reacts with nitrous acid to form aminoazo-benzene or Bismarck-brown, which is the cause of the colour obtained.

The colour in this method is only progressively developed ; and, in consequence, an attempt should not be made to compare the colours unless they have been standing for about thirty minutes.

(*b*) *Ilosvay's method of estimating nitrites.*

Solutions required : Weigh 0·1 gram of *a*-naphthylamine ; dissolve in 200 c.c. of dilute acetic acid (sp. gr. 1·04).

Dissolve 0·5 gram of sulphanilic acid in 200 c.c. of dilute acetic acid.

100 c.c. of the sample of water are placed in a Nessler glass ; and in another glass are placed 99 c.c. of distilled water and 1 c.c. of standard nitrite of potassium, as made in the last method. To each of these solutions add 2 c.c. of the sulphanilic acid solution and 2 c.c. of the naphthylamine solution. Allow to stand about five minutes and then compare the pink tints. If the tints are not equal then abstract a definite amount of the darker solution with a pipette, and make up to the original volume with distilled water. Suppose, for example, that the sample was darker, and that 40 c.c. were removed, and the bulk made up to the original volume of 100 c.c., when the tints were exactly the same ; then 60 c.c. of the original 100 c.c. were equal to 1 c.c. of the standard nitrite solution. Suppose 1 c.c.

of this solution = 0·00001 gram N_2O_3, the 60 c.c. of the sample are equivalent to 0·00001 gram N_2O_3, and therefore the amount of N_2O_3 in parts per 100,000 will be equal to

$$\frac{0{\cdot}00001 \times 100{,}000}{60} = 0{\cdot}0167 \text{ gram } N_2O_3$$

$$= 0{\cdot}0097 \text{ gram of nitrous nitrogen.}$$

It should be mentioned that a slight pink colour is acquired by the blank solution if it stands too long.

Nitrites should never be present in potable waters as they usually indicate insufficient oxidation of the organic matter, and indeed the actual presence of sewage; the presence of nitrites must, therefore, always be looked upon with the gravest suspicion. At the same time, it must not be forgotten that nitrites are often found in the waters from the Lower Greensand, the purity of which is unquestionable.

Hardness.

Solutions required:

Clark's soap solution. Weigh out

150 grams of lead plaster,
40 grams of dry potassium carbonate;

thoroughly mix in a mortar, add a little methylated spirit and continue the mixing until a uniform mass is obtained; add a little more methylated spirit and allow it to stand for some hours; decant off the clear liquid and dilute with a mixture of methylated spirit and distilled water (1 vol. of water to 2 vols. of methylated spirit) until exactly 14·25 c.c. of the solution are required to form a permanent lather with the standard calcium chloride solution.

Clark's soap solution may also be prepared by scraping 10 grams of shavings from a new cake of Castile soap, which are dissolved in 1000 c.c. of 35 per cent. alcohol, care being taken to dissolve all the soap; the solution is then filtered and kept tightly stoppered.

Standard calcium chloride solution. Weigh out 0·2 gram of pure Iceland Spar; dissolve it in dilute hydrochloric acid in a dish provided with a glass cover to prevent loss by spirting; evaporate the solution to dryness on the water bath;

redissolve the residue in distilled water and evaporate three times to remove all the hydrochloric acid ; and finally dissolve in water and make up to 1000 c.c.

Hardness is of two kinds, " Temporary Hardness," which is due to the solution in the water of the carbonates of calcium and magnesium, and which can be removed by boiling ; and " Permanent Hardness," which is primarily due to the presence of calcium and magnesium sulphates and which cannot be removed by boiling.

Estimation of total hardness. Measure out 50 c.c. of the water into a stoppered bottle of about 200 c.c. capacity, and run the soap solution from a burette into the water in the bottle, at first 0·5 c.c. at a time and finally 0·1 c.c. at a time, until, on shaking the bottle vigorously, a lather is formed which remains unbroken on the surface for three minutes ; the volume of the soap solution used is then read off on the burette. If more than 16 c.c. of the soap solution are used, 25 c.c. of the water should be diluted with distilled water, freed from carbon dioxide by boiling, and this diluted solution titrated as before. The degree of hardness is found by reference to the following table.

Table of hardness.

c.c. of soap solution used	$CaCO_3$ per 100,000	c.c. of soap solution used	$CaCO_3$ per 100,000	c.c. of soap solution used	$CaCO_3$ per 100,000
1·00	0·48	6·25	7·78	11·50	15·63
1·25	0·88	6·50	8·14	11·75	16·03
1·50	1·27	6·75	8·50	12·00	16·43
1·75	1·63	7·00	8·86	12·25	16·82
2·00	1·95	7·25	9·21	12·50	17·22
2·25	2·40	7·50	9·57	12·75	17·62
2·50	2·60	7·75	9·93	13·00	18·02
2·75	2·92	8·00	10·30	13·25	18·41
3·00	3·25	8·25	10·67	13·50	18·81
3·25	3·58	8·50	11·05	13·75	19·21
3·50	3·90	8·75	11·42	14·00	19·60
3·75	2·96	9·00	11·80	14·25	20·00
4·00	4·57	9·25	12·18	14·50	20·40
4·25	4·93	9·50	12·56	14·75	20·79
4·50	5·29	9·75	12·93	15·00	21·19
4·75	5·64	10·00	13·31	15·25	21·60
5·00	6·00	10·25	13·68	15·50	22·02
5·25	6·36	10·50	14·06	15·75	22·43
5·50	6·71	10·75	14·44	16·00	22·86
5·75	7·07	11·00	14·84		
6·00	7·43	11·25	15·22		

Estimation of permanent hardness. Place 100 c.c. of the water in a beaker and boil to about one-third of its volume; wash out the contents of the beaker into a 100 c.c. graduated flask; cool and make up to the 100 c.c. mark with distilled water, freed from carbon dioxide by boiling; the salts which produce temporary hardness are precipitated by the boiling and filtered off: titrate 50 c.c. of the filtered solution with the soap solution as before. The permanent hardness thus obtained is subtracted from the total hardness, the result being the temporary hardness.

Estimation of dissolved oxygen in waters.

For the methods employed in this determination see p. 46.

Poisonous metals.

The two poisonous metals of most importance are usually copper and lead; iron, zinc, tin, and arsenic come next in importance. To test for these metals qualitatively, the sample of water should be concentrated if necessary. Then place 50 c.c. of each in test glasses, add one drop of dilute hydrochloric acid, and then one drop of ammonium sulphide from the end of a glass rod; a brown colour will be produced from either lead, copper or iron; a white precipitate with zinc.

(1) When the colour is brown, divide the solution into two portions; to one portion add excess of hydrochloric acid; the colour will disappear if it is iron. Confirm the iron by taking a fresh portion of the concentrated solution, add a drop of nitric acid, and then a few drops of potassium ferrocyanide, when a blue colour will be produced.

(2) If it is not iron, take the other portion, add a few drops of potassium cyanide; if the colour is destroyed, it is copper. Confirm the copper by taking two fresh portions of the concentrated solution; to one portion add potassium ferrocyanide, when a bronze colour or precipitate is produced if it is copper; and to the other portion add ammonia, when a blue colour results which confirms copper.

(3) If the precipitate does not dissolve when potassium cyanide is added, it is very probably lead. To confirm lead,

take two fresh portions of the concentrated solution; to one add a drop or two of clear potassium iodide, when a yellow precipitate or opalescence of lead iodide will appear; and to the other portion add potassium chromate solution which produces a yellow precipitate of lead chromate: thus confirming lead.

(4) If the precipitate is white, it may be caused by zinc. This may be confirmed by adding potassium ferrocyanide to a portion of the concentrated solution, when a white gelatinous precipitate is produced.

(5) When arsenic is in solution the colour of the sample of water becomes yellow on the addition of one drop of ammonium sulphide; a yellow precipitate may be produced if the arsenic is in sufficient quantity. It may be confirmed by placing some zinc foil in a large test-tube, and boiling it with sodium hydroxide; a few drops of the concentrated water containing the dissolved arsenic compound are added and boiled again; at the same time, hold a piece of filter paper over the mouth of the test-tube, on which there is a spot of silver nitrate, and the latter becomes black or brown if arsenic is present. A blank experiment should be done with the reagents themselves, because the zinc may contain arsenic.

(6) If it is thought that tin is present in the water, the concentrated sample is reduced from the stannic to the stannous condition by adding some metallic copper and a little hydrochloric acid and boil well; then a few drops of mercuric chloride, when a silky precipitate of mercurous chloride is produced, which may be further reduced to dark grey mercury, if tin is dissolved in the water.

Estimation of poisonous metals.

Lead.

Standard lead solution. Weigh out 0·183 gram of pure lead acetate; dissolve in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 0·0001 gram of lead.

Saturated sulphuretted hydrogen solution This solution is obtained by passing hydrogen sulphide gas, from a Kipp's apparatus, into distilled water to saturation.

For the estimation, place 50 or 100 c.c. of the water in a Nessler cylinder and add to it six drops of acetic acid and then two drops of the saturated sulphuretted hydrogen solution; in the presence of lead a brown or black colour will be produced; or one drop of ammonium sulphide may be used instead. If the colour is obtained, place in another cylinder a measured quantity of the standard lead solution (0·5, 1·0, 1·5 c.c., etc.); fill the cylinder with distilled water to the 50 or 100 c.c. mark; add acetic acid and sulphuretted hydrogen solution, or ammonium sulphide, as before, and match the colours in the two cylinders.

Copper.

Standard copper solution. Weigh out 0·393 gram of pure crystallized copper sulphate; dissolve in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 0·0001 gram of copper.

A very dilute solution of potassium ferrocyanide.

Concentrate 1000 c.c. of the water, if necessary, by evaporation on the water bath to 100 c.c. and place it in a Nessler cylinder; and to the water add six drops of acetic acid and three drops of the potassium ferrocyanide solution; in the presence of copper a brown colour is obtained. It may happen that the colour will be produced in the water without any concentration: in which case, time will be saved. The brown colour is matched by treating measured volumes of the standard copper solution in another cylinder in a similar manner; and from the number of c.c. of the standard copper solution required, the amount of copper present in the sample is calculated.

Iron.

Standard iron solution. Weigh out 0·497 gram of pure crystallized ferrous sulphate; dissolve in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 0·0001 gram of iron.

Evaporate 1 litre of the sample of water, if necessary, to 100 c.c.; place in a Nessler glass: add two or three drops of acetic acid, and then a drop of saturated sulphuretted

hydrogen, a dark colour will be produced of ferrous sulphide. Place in another Nessler glass a measured quantity of the standard iron solution and repeat the process in the same way, and match the colours as before. The amount of iron can then be calculated.

Mineral analysis.

Determination of alkalinity. Measure out 500 c.c. of the water into a large flask; add a few drops of methyl orange as an indicator; and run in from a burette N/10 hydrochloric acid until the yellow colour of the methyl orange just turns to pink; the number of c.c. of N/10 acid used multiplied by 0·005 gives the alkalinity in terms of calcium carbonate.

Estimation of silica. One litre of the water is placed in a large porcelain dish with about 5 c.c. of concentrated hydrochloric acid; the whole is evaporated to small bulk on a water bath; the contents of the dish are then washed out into a platinum dish and evaporated to dryness; the residue is dried in the air oven. The dry residue is moistened with a few drops of concentrated hydrochloric acid; the acid is then evaporated on the water bath and the residue placed in an air oven at 150° C. for 30 minutes. After being in the air oven for 30 minutes, the residue is treated with water, filtered, and the filter well washed with hot water and dried in a water oven. The dried filter paper and residue are placed in a platinum dish and ignited over a Bunsen flame; as soon as a white or nearly white ash is obtained, the dish is placed in a desiccator, allowed to cool and weighed; the increase in weight of the dish less the weight of the filter paper ash represents silica.

Estimation of iron. To the filtrate from the silica determination, add a few drops of bromine and a small quantity of solid ammonium chloride, make alkaline with ammonia, boil and filter, well wash the filter with hot water, dry the filter paper with the iron oxide precipitate in the water oven, and when dry ignite it in a platinum dish and weigh the residue. The weight of the residue less the weight of the filter paper ash represents iron as Fe_2O_3.

Estimation of lime. If the filtrate from the iron determination is large, it must first be concentrated to about 250 c.c.;

after this concentration, the solution is made alkaline with ammonia, and about 0·5 gram of solid ammonium oxalate is added, the whole is then heated to boiling and then allowed to stand till cool. As soon as the solution is cool, it is filtered and the precipitate is well washed with hot water and dried in a water oven; when dry the filter paper and precipitate are placed in a platinum dish and ignited over a blowpipe till the weight is constant; the increase in weight, less the weight of the filter paper ash, is calculated as CaO.

Estimation of magnesia. The filtrate from the lime determination is evaporated to small bulk and made strongly alkaline with ammonia, and 0·5 gram of solid ammonium phosphate added, and the solution stirred with a glass rod until a precipitate begins to appear; as soon as a precipitate begins to appear, the beaker is covered with a clock glass, set aside and allowed to stand overnight. On the following morning, the precipitate is filtered off; well washed with water containing ammonia, and dried in a water oven; when dry, the precipitate and filter paper are placed in a platinum dish and ignited first over a Bunsen flame and finally over a blowpipe till constant in weight. From the increase in weight of the platinum dish, the weight of the ash of the filter paper is subtracted, the remainder is magnesium pyrophosphate ($Mg_2P_2O_7$). The weight of magnesium pyrophosphate obtained multiplied by 0·3604 gives the weight of magnesia.

Estimation of alkalies. Measure out 500 c.c. of the water into a porcelain dish and evaporate it to dryness on a water bath; dissolve the residue in a small quantity of distilled water and filter; the filtrate is placed in a glass dish, three drops of dilute sulphuric acid are added and the whole is evaporated to dryness; the residue is dissolved in a small quantity of distilled water, two drops of barium chloride solution added, followed by excess of baryta water, again evaporated to dryness and allowed to stand for 30 minutes. After standing, the residue is dissolved in 50 c.c. of distilled water and filtered; the excess of barium salt in the filtrate is then precipitated with excess of ammonium carbonate; the precipitate filtered off and the filtrate evaporated to dryness and heated to 150° C. to remove

the ammonium salts. When the ammonium salts have been removed, the residue is dissolved in distilled water and filtered ; the filtrate is placed in a weighed platinum dish, a few drops of hydrochloric acid added, and evaporated to dryness, dried in the water oven and weighed ; the residue consists of potassium and sodium chlorides. The weighed residue of the mixed chlorides is dissolved in water, a few drops of hydrochloric acid added and an excess of a solution of platinum chloride ; the solution is then evaporated to a syrupy consistency on a water bath in a porcelain basin ; the contents of the basin are allowed to cool and are then treated with alcohol (specific gravity, 0·864), washed by decantation and the washings passed through a dried and weighed filter paper ; the washing is continued until the alcohol is colourless ; the precipitate is finally collected on the filter paper, washed with alcohol, dried in the water oven and weighed. The weight of potassium platino-chloride obtained multiplied by 0·3094 gives the weight of potassium chloride ; the weight of sodium chloride being obtained by subtracting this from the weight of the mixed chlorides.

Estimation of sulphates. Measure out 500 c.c. of the water into a beaker of about 800 c.c. capacity ; place the beaker on a sand bath and evaporate the water to about 150 c.c. ; and to the water while still hot, add 10 c.c. of concentrated hydrochloric acid and 25 c.c. of a boiling solution of barium chloride ; continue the boiling for a short time, and allow the precipitate to settle in the beaker. As soon as the precipitate has settled, collect it on a filter paper, specially prepared to retain barium sulphate ; wash with hot water until the washings give no reaction for chlorides, and dry in a water oven ; when the precipitate is dry, carefully transfer it from the filter paper to a piece of glazed paper and cover it with a watch glass ; fold up the filter paper and incinerate it in a platinum dish over a Bunsen flame ; after the incineration of the paper, carefully brush the precipitate into the dish and again ignite over the Bunsen flame ; allow the dish to cool in a desiccator, and weigh ; from this weight subtract the weight of the filter paper ash. The weight of barium sulphate obtained multiplied by 0·34293 gives the weight of sulphate (as SO_3).

Interpretation of results.

The following general rules may be mentioned, but the interpretation of analyses is illustrated later by some typical examples.

Deep well water. A deep well water containing over 0·006 parts of albuminoid ammonia per 100,000 is suspicious, as this amount would indicate surface pollution. The nitrates in a deep well water may be disregarded unless they are present in excessive quantities with a low albuminoid ammonia. The oxygen absorbed should be low. The chlorine should be low unless the well is near a coal seam, or near to the sea or a brackish estuary. Nitrites may be produced by reduction of nitrate, as in the water from the Lower Greensand, but they must be looked upon with suspicion.

Shallow well water. If the amount of albuminoid ammonia and oxygen absorbed in a shallow well water are high, and there is no other indication of pollution, the organic matter present may be of vegetable origin. High nitrate and chlorine, whether or not accompanied by high free and albuminoid ammonias, undoubtedly point to past pollution with animal organic matter.

Moorland waters. Moorland waters are often yellowish brown in colour. Albuminoid ammonia and oxygen absorbed are usually high with a low nitrate, pointing to organic matter of vegetable origin. The reaction of these waters is of very great importance, as, if they are even slightly acid, they will probably have a plumbo-solvent action.

The proportion of total solids in a water is not of very great importance from a Public Health point of view; as it depends on the geological nature of the strata through which the water has passed.

Examples:

Free and saline ammonia. 500 c.c. of the sample were taken for the estimation, the first distillate was matched with 0·8 c.c. of the standard dilute ammonia solution, the second distillate with 0·2 c.c., the third distillate was free from ammonia.

0·8 + 0·2 = 1·0 c.c. of the standard dilute ammonia solution used. Therefore the free ammonia in the sample

= 1·0 × 2 × 100 × 0·0001 = 0·02 part per 100,000.

Albuminoid ammonia. The first distillate gave a colour equal to 0·5 c.c. of the standard dilute ammonia solution, the second to 0·2 c.c., the third distillate was free from ammonia.

0·5 + 0·2 = 0·7 c.c. of the standard dilute ammonium chloride solution used.

0·7 × 2 × 100 × 0·0001 = 0·014 part per 100,000 of albuminoid ammonia.

Total solid matter. 100 c.c. of the water were taken for the estimation.

The weight of the dish and solid matter was 34·444 grams.
The weight of the dish was .. .. 34·431 grams.
∴ The weight of the solid matter was .. ·013 gram.

0·013 × 1000 = 13·0 parts per 100,000 of total solid matter.

Chlorides. 50 c.c. of a sample were titrated with the standard silver nitrate solution, and required 4·5 c.c. to produce a slight permanent red colour, using the potassium chromate solution as an indicator.

The amount of chlorine as chlorides in the sample therefore was 4·5 parts per 100,000.

Oxygen absorbed from potassium permanganate solution. 250 c.c. of a sample were used for an estimation, 10 c.c. of the standard potassium permanganate solution were added and at the end of four hours 8·7 c.c. of the standard oxalic acid solution were required to decolourize the solution.

The amount of potassium permanganate solution used by the sample was 10·0 − 8·7 = 1·3 c.c.

1·3 × 0·0001 × 4 × 100 = 0·052 part per 100,000 of oxygen absorbed.

Nitrates. (*a*) *Gasometric.* 100 c.c. of a sample were used for the estimation and the volume of gas obtained was 0·7 c.c. The temperature was 16° C. and the pressure 770 mm.

The volume of gas reduced to normal temperature and pressure

$$= \frac{0{\cdot}7 \times 273 \times 770}{289 \times 760} = 0{\cdot}67 \text{ c.c.}$$

1 c.c. of NO weighs 0·00134 gram.

Therefore $0{\cdot}67 \times 0{\cdot}00134 \times 1000 = 0{\cdot}898$ part per 100,000 of nitrogen as nitrate.

(*b*) *Colourimetric.* In a colourimetric determination it was found that the standard potassium nitrate solution gave a very much deeper colour than the water, and that 10 c.c. of the standard solution was equal in tint to the 100 c.c. of the water.

Therefore the nitrogen as nitrate in the 25 c.c. of water taken

$$= \frac{5 \times 10}{100} \times 0{\cdot}0001 \times 4000 = 0{\cdot}2 \text{ part per } 100{,}000.$$

(*c*) *Ammonia method.* The first distillate was matched by 4·7 c.c. of the standard solution, the second distillate by 0·5, the third by 0·1 c.c., the fourth distillate contained no ammonia.

$4{\cdot}7 + 0{\cdot}5 + 0{\cdot}1 = 5{\cdot}3$ c.c. of the standard solution used.

Now 17 grams of ammonia contain 14 grams of nitrogen, therefore the nitrogen as nitrate in the sample was :

$$\frac{5{\cdot}3 \times 14}{17} \times 0{\cdot}0001 \times 4 \times 100 = 0{\cdot}174 \text{ part per } 100{,}000.$$

Nitrites. In an estimation of nitrites, 100 c.c. of the water gave a colour which was exactly matched with 0·5 c.c. of the standard nitrite solution.

The amount of nitrite as N_2O_3 therefore was

$$0{\cdot}5 \times 0{\cdot}00001 \times 1000 = 0{\cdot}005 \text{ part per } 100{,}000.$$

Lead. The colour obtained with 100 c.c. of the water was exactly matched with 15 c.c. of the standard lead solution.

The amount of lead in the water was

$$1{\cdot}5 \times 0{\cdot}0001 \times 1000 = 0{\cdot}15 \text{ part per } 100{,}000.$$

Copper. 1000 c.c. of water after concentration to 100 c.c. gave a colour which was matched with 0·7 c.c. of the standard copper solution.

The amount of copper in the water was

$$0{\cdot}7 \times 0{\cdot}0001 \times 100 = 0{\cdot}007 \text{ part per } 100{,}000.$$

Typical analyses.

The following typical analyses are introduced to show the wide differences to be met with both in the inorganic and organic constituents of waters. The numbers are in parts per 100,000 except where specially mentioned. It should also be remembered that an analysis of the organic constituents need not be the final judgment of a water. It is necessary that the chemist should know some of the conditions to aid him in a sound judgment of the results of his analysis[1]. For this purpose (1) he should be acquainted with the geological nature of the district from which the water is obtained; and the general saline constituents of the geological strata, for an analysis may show high chlorides which have been dissolved from the rocks. This is particularly the case with wells near the sea coast and from the Lower Greensand rocks; (2) he should be acquainted with the gathering ground, the methods of storage and distribution of the water; (3) if the water is obtained from wells, he should visit the place himself to note any faults in the outlet, the surface drainage and in the covers of the wells; (4) he should know the amount of the rainfall before and after the analysis was made, for after a heavy rainfall the analysis often gives results which differ very con-

Physical characters	(1) Well aerated, no smell or sediment, reaction slightly alkaline	(2) Slightly opalescent, but there was no smell.
Free and saline ammonia ..	0·001	0·0860
Albuminoid ammonia	0·002	0·0055
Oxygen absorbed from potassium permanganate in four hours at 80° F.	0·007	0·0060
Chlorine	2·500	9·8000
Nitric nitrogen	0·600	nil
Nitrous nitrogen	nil	0·0050
Total solids	44	81
(*a*) Volatile solids	22	15
(*b*) Non-volatile solids (faint charring on incineration)	22	66 (faint charring on heating)
Total hardness (per 100 c.c.) ..	17°	11°
(*a*) Permanent hardness ..	5°	10·5°
(*b*) Temporary hardness ..	12°	0·5° (9 parts of iron per 100,000 were found)

[1] See Purvis, *Journ. Roy. Sany. Inst.* 1905, vol. XXVI, p. 429.

siderably from one before the rainfall; and (5) in case of any doubt a bacterial analysis should be undertaken. A water may be chemically pure and still contain the germs of disease; and, on the other hand, a water may be bacterially pure, whereas chemically the constituents might indicate contamination.

Number (1) is a water obtained from a deep well in the Upper Chalk. Organically it is very pure; the only fault is the high degree of hardness, which could easily be remedied, as it is chiefly caused by the acid carbonate of calcium and in a less degree of the salt of magnesium.

Number (2) is a water from the Lower Greensand. The high free ammonia and chlorine are noticeable: high figures are always obtained from such waters; but the albuminoid ammonia is comparatively low, and the figure for oxygen absorbed is also low: the absence of nitric nitrogen should be noticed, and the presence of nitrous nitrogen. The latter must not condemn the water. It has not been produced by bacteria, but by the reduction of the nitrates as a consequence of the iron salt to which the colour of the Lower Greensand owes its origin being partly oxidised to the ferric condition.

	(3)	(4)
Physical characters	Clear, no smell, no sediment, neutral reaction	Slightly opalescent, no smell, light yellow brown colour
Free and saline ammonia ..	0·0010	0·0720
Albuminoid ammonia	0·0024	0·0240
Oxygen absorbed from potassium permanganate in three hours at 80° F.	0·0040	0·0170
Chlorine	4·0000	18·8000
Nitric nitrogen	0·7500	2·000
Nitrous nitrogen	nil	0·004
Total solids	46	128
(*a*) Volatile solids	10	45
(*b*) Non-volatile solids ..	36 (no charring on heating)	83 (blackened on heating)
Total hardness (per 100 c.c.) ..	22°	18°
(*a*) Permanent hardness ..	10°	12°
(*b*) Temporary hardness ..	12°	6°
	No poisonous metals	No poisonous metals

Number (3) is a water from a shallow well in the Magnesian Limestone. The outstanding figures are those for the chlorine and nitric nitrogen. The two ammonias and the oxygen absorbed figures are low. The water shows, therefore, signs of

past contamination, and the potential danger is obvious. Such a well would have to be carefully watched, and should be bacterially analysed. Care should be taken to protect the water from any possible contamination.

Number (4) is a water from a shallow well. All the numbers are very high; and it must be condemned as quite unfit for drinking purposes. It shows unmistakeable signs of a high degree of pollution.

	(5)	(6)
Physical characters	Brown colour, sediment, slightly acid reaction, no smell	Good
Free and saline ammonia ..	0·0960	trace
Albuminoid ammonia	0·0470	0·0574
Oxygen absorbed from potassium permanganate in three hours at 80° F.	0·3950	0·0050
Chlorine	3·2000	1·2000
Nitric nitrogen	trace	0·5000
Nitrous nitrogen	nil	nil
Total solids	22	31
(*a*) Volatile solids	14	12
(*b*) Non-volatile solids ..	8 (blackened on heating)	19 (very slight charring on heating)
Total hardness (per 100 c.c.) ..	6·5°	16°
(*a*) Permanent hardness ..	3·5°	4·5°
(*b*) Temporary hardness ..	3·0°	11·5°
	No poisonous metals	No poisonous metals

Number (5) is a water from a village pond and has been used for drinking purposes. The pollution was partly caused by vegetable growths; but a microscopic examination of the sediment showed the bodies and wings of various insects as well as vegetable cells and debris. The water must be wholly condemned for drinking purposes. The softness of the water is noticeable.

Number (6) is a water from a well in the Upper Chalk of about 37 feet deep. The two outstanding figures are those for the albuminoid ammonia and the nitric nitrogen. The former is a little disconcerting; but the low chlorine and oxygen absorbed figures are in its favour. On the whole, the water must be watched carefully, and a bacterial analysis should be made. Care should also be taken that the mouth of the well is protected.

Physical characters	(7) Neutral reaction, clear, no sediment	(8) Neutral reaction, no smell, no sediment, clear
Free and saline ammonia ..	0·0420	0·0020
Albuminoid ammonia	0·2940	0·0100
Oxygen absorbed from potassium permanganate in three hours at 80° F.	0·1880	0·3000
Chlorine	32·0000	6·000
Nitric nitrogen	3·0000	7·000
Nitrous nitrogen	0·0090	nil
Total solids	255	—
(*a*) Volatile solids	50	—
(*b*) Non-volatile solids ..	205 (blackened on heating	—
Total hardness (per 100 c.c.) ..	34°	33°
(*a*) Permanent hardness ..	31°	—
(*b*) Temporary hardness ..	3°	—
	No poisonous metals	No poisonous metals

Number (7) is water from a shallow well. It is highly polluted with organic matter. Every item is high and the presence of nitrous nitrogen shows that bacterial action is going on. It must be condemned as a dangerous water: the high degree of hardness is also unfavourable to its use for other purposes.

Number (8) is also from a shallow well. All the figures are high except the free ammonia. The high nitrate and chlorine figures show considerable past pollution: and the high figures for the albuminoid ammonia and the oxygen absorbed show that the pollution is going on. It must be condemned for drinking purposes.

Physical characters	(9) Not very clear, poorly ærated neutral reaction	(10) Slightly turbid, pale yellow colour (grains per gallon)
Free and saline ammonia ..	0·0060	none
Albuminoid ammonia	0·0056	·0217
Oxygen absorbed from potassium permanganate in four hours at 80° F.	0·0080	·1092
Chlorine	3·1000	9·200
Nitric nitrogen	3·5000	1·600
Nitrous nitrogen	nil	nil
Total solids	60	127
(*a*) Volatile solids	14	—
(*b*) Non-volatile solids ..	46 (slight charring on heating)	—
Total hardness (per 100 c.c.) ..	20°	—
(*a*) Permanent hardness ..	7°	—
(*b*) Temporary hardness ..	13°	—
	No poisonous metals	A microscopic examination of the slight deposit showed that it was organic matter

Number (9) is from a shallow well (20 feet deep) in the Chalk formation: it shows definite signs of past contamination, as indicated by the high nitric nitrogen and the comparatively high chlorine figure; the two ammonias and the oxygen absorbed figures are also higher than is usually found in deep wells from the Chalk; and for these reasons also it must be condemned.

Number (10) is from a well in the gravel above the Chalk. The numbers in No. 10 are in grains per gallon. With the exception of the free ammonia, all the constituents are high; and both the smell on heating and the microscopic examination of the sediment showed that the water was polluted with sewage, and was unfit for drinking purposes. We are indebted to Mr West Knights for this analysis.

	(11)	(12)	(13)	(14)	(15)
Free and saline ammonia ..	0·000	0·557	0·006	0·003	0·024
Albuminoid ammonia	0·004	0·550	0·008	0·004	0·004
Oxygen absorbed from potassium permanganate in four hours at 80° F.	0·170	0·871	—	—	—
Chlorine	0·800	3·570	8·700	6·800	22·95
Nitric nitrogen	nil	nil	6·350	3·800	0·220
Nitrous nitrogen	nil	nil	—	—	—
Total solids	6·000	42·5	112·5	220·2	194·0

Number (11) is an analysis of the Birmingham City Water Supply by Mr J. F. Liverseege, F.I.C., City Analyst. The gathering ground is near Rhayader in Wales; the geological formation is Silurian and consists of shales and flagstones, with some massive layers of conglomerate. None of the land is cultivated; the whole is occupied as rough hill pasture restricted to sheep farming; the water shows a complete absence of free and saline ammonia, low chlorine, and total solids and medium oxygen absorbed. (For the mineral analysis of Number (11) see p. 33.)

Number (12) is an analysis of a water from a shallow well with a cesspool at a short distance from it; the free and saline and albuminoid ammonias and the oxygen absorbed are exceedingly high; the water is grossly contaminated with fresh animal organic matter and must be condemned.

Numbers (13), (14) and (15) are analyses of Burton-on-Trent waters by Messrs F. E. Lott and C. G. Matthews (*Jour. Soc.*

Chem. Ind. 1911, vol. xxx, p. 69). No. (13) consists partly of rain water in the Trent valley and partly of water from springs in the Marl bed underlying the gravel and shows a high nitrate, total solids and chlorine. No. (14) is from a bore hole in the Marl bed and No. (15) is from a bore hole in the Lower Keuper Sandstone and shows high chlorine, total solids, and free and saline ammonia, with a low nitrate. For the mineral analyses of numbers (13), (14) and (15) see p. 34.

	(16)	(17)	(18)
Free and saline ammonia	0·001	0·000	0·003
Albuminoid ammonia	0·001	0·005	0·002
Oxygen absorbed from potassium permanganate in four hours at 80° F.	—	0·107	0·002
Chlorine	1·800	1·420	2·230
Nitric nitrogen	0·250	0·110	0·300
Nitrous nitrogen	—	—	—
Total solids	30·000	28·250	16·200
Mineral solid matter	—	20·750	—
Organic solid matter	—	7·500	—

Number (16) is an analysis by Mr C. L. L. Claremont of the Portsmouth Public Water Supply; it is taken from a deep chalk spring in the western end of the South Downs; every item is low and the water is satisfactory. For the mineral analysis of number (16) see p. 35.

Number (17) is an analysis by Mr E. Russell, B.Sc., F.I.C., City Analyst, of the Bristol City Water Supply showing a medium oxygen absorbed and a moderately high organic solid matter.

Number (18) is an analysis by the late Professor J. Campbell Brown of a water from a deep bore hole in the Keuper Sandstone in south-west Lancashire; every item is low and the water is satisfactory.

	(19)	(20)	(21)	(22)
Free and saline ammonia	0·003	0·006	0·0006	traces
Albuminoid ammonia	0·009	0·017	0·0052	0·0034
Oxygen absorbed from potassium permanganate in four hours at 80° F.	0·840	0·296	0·1294	0·1150
Chlorine	1·500	0·600	0·9000	1·3000
Nitric nitrogen	trace	0·109	0·0066	0·0500
Nitrous nitrogen	—	—	—	—
Total solids	10·160	3·980	3·800	7·000
Mineral solids	—	—	3·080	4·600
Organic and volatile solids	—	—	·720	2·400
Total hardness	—	—	·630	3·000

Numbers (19) and (20) are analyses of the Liverpool City Water Supply by the late Professor J. Campbell Brown (*Engineering*, 1903, LXXVI, 472), Number (19) is from the Rivingstone Supply in the Millstone Grit formation and number (20) from the Vyrnwy Supply. Both waters are quite satisfactory.

Number (21) is an analysis of the Glasgow Water Supply from Loch Katrine by Mr F. W. Harris, City Analyst. All the items are extremely low. The total solids and the hardness are low. The obvious drawback to the water is its great softness. (See p. 36 for the mineral analysis.)

Number (22) is an analysis of the water supply of the County Borough of Blackpool which C. Arthur, Esq., secretary to the Fylde Water Board, has kindly placed at our disposal. The water is an upland surface water, the gathering ground of which is the Lancashire and Yorkshire Moors. The surface is soft and peaty and is thoroughly safeguarded from all risks of animal pollution. The water is exceedingly soft; there is no temporary hardness; and the hardness is wholly "permanent," and is only 3°; the water is so soft that it has an erosive action on lead pipes; it is slightly brown and turbid.

	(23)	(24)	(25)
Free and saline ammonia	0·004	0·005	0·002
Albuminoid ammonia	0·005	0·005	0·002
Oxygen absorbed from potassium permanganate in four hours at 80° F.	0·006	—	0·004
Chlorine	2·000	1·800	4·250
Nitric nitrogen	0·371	0·350	0·173
Nitrous nitrogen	—	—	—
Total solids	18·600	17·200	27·600

Numbers (23), (24) and (25) are analyses by the late Professor J. Campbell Brown (*Engineering*, 1903, LXXVI, 472) of waters from the same bore hole at Newton in south-west Lancashire at different depths, number (23) at 60 ft., number (24) at 130 ft., number (25) at 500 ft. The particular items to be noticed are the decrease in the free and saline and albuminoid ammonias and the nitrate, and the increase in the total solids and chlorine, as the depth increases.

Numbers (26), (27), (28), (29) and (30) are analyses by Mr J. White, F.I.C., County Analyst of Derbyshire, of samples of water taken from a bore hole at Ilkeston in Derbyshire.

	(26)	(27)	(28)	(29)	(30)
Free and saline ammonia ..	0·060	0·875	0·120	0·100	0·080
Albuminoid ammonia ..	0·002	0·004	0·006	0·008	0·004
Oxygen absorbed from potassium permanganate in four hours at 80° F. ..	0·036	0·680	—	0·124	0·035
Chlorine	17·1	1491·0	142·0	10·7	4·8
Nitric nitrogen	0·0	0·0	0·0	0·055	0·055
Nitrous nitrogen	—	—	—	—	—
Total solids	59·5	2628·0	282·0	62·5	51·0

Number (26) is the water originally yielded by the bore hole, number (27) is an abnormal water which gained access to the bore hole by the splitting of the steel lining tubes. Number (28) is a sample collected from the pumping shaft after the abnormal water had appeared. Numbers (29) and (30) are from the pumping shaft about 80 ft. below the surface before the water was found.

The particular items to note are the enormous chlorine, total solids and free and saline ammonia in the abnormal water, and the mineral analysis of this water should be noted (p. 35).

	(31)	(32)	(33)	(34)
Physical characters	Yellow, turbid, no smell	Deep yellow, slightly turbid, no smell	Deep yellow, turbid, no smell	Slightly turbid, no smell
Free and saline ammonia	none	·0007	0·0028	trace
Albuminoid ammonia ..	·0112	·0371	·0119	·0035
Oxgyen absorbed from potassium permanganate in 15 *minutes at* 140° F.	·0616	·1680	·1624	·0252
Chlorine	3·400	11·400	7·700	1·800
Nitric nitrogen	1·100	4·000	1·500	·500
Total solids	44·00	96·00	87·00	30·00
	The sediment showed earthy matter on a microscopic examination	There was a deposit of organic matter	There was a deposit of organic matter	A slight deposit of oxide of iron

The above analyses are kindly supplied by Mr J. West Knights, Public Analyst for Cambridge, and the numbers represent grains per gallon.

Number (31) is from a shallow well and shows that the albuminoid ammonia and oxygen absorbed figures are high; the total solids and chlorides are not very low; the nitrates are high and unsatisfactory; the microscopic examination was bad. The water was considered to be polluted with sewage, and unfit for drinking purposes.

Number (32) is also from a well and shows that the total solids, chlorides and nitrates all very high; the high nitrate figure must be specially noticed: the albuminoid ammonia and the oxygen absorbed figures are also very high. The water was condemned as highly polluted with sewage and quite unfit for drinking purposes.

Number (33) is also from a shallow well. The total solids, chlorides and nitrates are very high and unsatisfactory; the albuminoid ammonia and the oxygen absorbed are both very high, and show the presence of much organic matter; the appearance and the microscopic examination were also bad. The water was considered to be polluted and unfit for drinking purposes.

Number (34) is from a well in the Chalk. The solids, chlorides and nitrates are fairly low and quite satisfactory; the two ammonias and the oxygen absorbed are all low and show the absence of organic matter. The water is unpolluted and fit for drinking purposes.

	(35)	(36)	(37)	(38)
Physical characters	Slightly turbid, no smell	Clear, no smell, no deposit	Very turbid, no smell	Clear, no smell
Free and saline ammonia	0·0007	0·0005	·0357	0·0238
Albuminoid ammonia ..	·0021	0·0005	·0014	·0007
Oxygen absorbed from potassium permanganate in 15 *minutes at* 140° F.	·0168	0·0084	·0140	·0112
Chlorine	2·000	1·500	5·800	1·600
Nitric nitrogen	·100	none	none	none
Total solids	28·00	24·00	65·00	18·00
	The slight turbidity was from a slight deposit of chalk		The turbidity was caused by a deposit of hydrated oxide of iron	

The above analyses have also been supplied by Mr West Knights. They show the differences between the water of the Chalk and the Lower Greensand as well as the differences in the water from different parts of the Greensand. The numbers represent grains per gallon.

Number (35) is from a deep well in the Chalk, and shows that the solids, chlorides and nitrates are low and quite satisfactory; the two ammonias and the oxygen absorbed figures

are all low, and show the absence of organic matter. The water was unpolluted and fit for drinking purposes.

Number (36) is from the Lower Greensand. The solids, chlorides, nitrates, the two ammonias and the oxygen absorbed are all very low. The water was unpolluted and quite fit for drinking purposes.

Number (37) is also from the Greensand. All the items are low: and the water is quite unpolluted. The deposit of the oxide of iron could be cleared by subsidence or filtration.

Number (38) is also from the Greensand. All the items are low, and the water is quite fit for drinking purposes.

Mineral Analyses. The following are a series of analyses of the mineral constituents of various waters. They show the great differences there are in waters from different localities. The numbers represent parts per 100,000.

	(1)
Carbonates (as CO_3)	1·44
Sulphates (as SO_4)	0·10
Chlorides (as Cl)	0·95
Silicates (as SiO_4)	0·43
Calcium (as Ca)	1·03
Magnesium (as Mg)	0·18
Iron (as Fe)	0·08
Potassium (as K)	0·14
Sodium (as Na)	0·53

The above, No. (1), supplied by Mr J. F. Liverseege, City Analyst, Birmingham, is a mineral analysis of number (11) (see the organic analysis, p. 28) and is an upland surface water from the Silurian formation. Every item is low.

	(2)
Calcium carbonate..	25·00
Magnesium carbonate	9·80
Sodium sulphate	4·27
Sodium chloride	4·33

The water, No. (2), of which the above is the analysis of the dissolved salts, was taken from the Red Sandstone of Manchester. It is particularly interesting owing to the complete absence of calcium and magnesium sulphates. The sample also contained a very large amount of free carbon dioxide; and it was so large that unless the containing bottles had been immediately corked a large proportion of it would have escaped.

The above analysis is published by permission of T. R. Wollaston, Esq.

	(3)
Sulphates (as SO_3)	19·71
Chlorine (as Cl)	9·57
Nitrogen (as nitrate)	0·20
Lime (as $CaCO_3$)	24·62
Magnesium (as MgO)	0·38
Potassium (as K_2O)	0·54
Sodium (as Na_2O)	25·50
Silica (as SiO_2)	0·14
Alkalinity (as Na_2CO_3)	15·10

The above, No. (3), is an analysis of an alkaline water. The interesting points in this water are its high alkalinity, the small quantity of magnesium salts, and the appearance of the lime as carbonate with the consequent absence of permanent hardness.

	(4)	(5)	(6)
Magnesium carbonate	4·9	3·1	3·6
Magnesium sulphate	17·74	32·0	19·5
Magnesium nitrate	8·7	5·2	0·3
Magnesium chloride	1·9	nil	nil
Calcium carbonate	20·2	20·0	17·8
Calcium sulphate	46·1	147·0	89·5
Sodium sulphate	nil	1·0	23·5
Potassium and sodium chlorides	12·0	11·2	37·8
Iron, silica and alumina	1·3	0·7	2·0

The above are the analyses of numbers (13), (14) and (15) (see the organic analyses, p. 28). They are due to Messrs Lott and Matthews. Number (4) is partly rain water from the Trent Valley and partly water from springs in the Marl bed underlying the gravel. Number (5) is from a bore hole in the Marl bed and number (6) from a bore hole in the Lower Keuper sandstone. The proportion of calcium sulphate is high, especially in numbers (5) and (6). Number (6) shows the presence of sodium sulphate in moderately large quantity.

	(7)
Magnesium carbonate	56·7
Magnesium sulphate	1·1
Magnesium chloride	24·0
Calcium carbonate	203·4
Potassium sulphate	4·3
Sodium chloride	34·3
Ferrous carbonate	1·5
Silica	14·5

Number (7) is an analysis by Muspratt of the water from the St Ann's well at Buxton and shows very high calcium carbonate, magnesium carbonate, sodium chloride and magnesium chloride. Note also the presence of ferrous carbonate. On being exposed to the air this water deposits hydrated ferric oxide, and it can be used as a natural purifying agent for the treatment of the Buxton sewage.

	(8)
Nitrogen as nitrates	0·25
Carbonates (as CO_2)	9·94
Sulphates (as SO_3)	0·48
Silica (as SiO_2)	0·60
Iron and alumina	0·30
Lime (as CaO)	12·80
Magnesia (as MgO)	0·50
Sodium (as Na)	0·31
Potassium (as K)	1·04

Number (8) is the analysis of the water number (16) (see organic analysis, p. 29) of the Portsmouth Public Water Supply from a deep chalk spring in the western end of the South Downs. The analysis is by Mr C. L. L. Claremont.

	(9)	(10)
Nitrogen as nitrate ..	trace	0·109
Alkalis	1·06	0·518
Magnesia	0·461	0·127
Lime	1·735	0·706
Iron	0·016	0·99
Oxide of manganese	0·017	0·059
Sulphates (as SO_3) ..	2·144	0·600
Silica	0·463	0·214

Numbers (9) and (10) are the analyses by the late Professor Campbell Brown of the Liverpool City Supply (see organic analyses, numbers (19) and (20), p. 29). Number (9) is from the Rivingstone supply in the Millstone Grit and number (10) from the Vyrnwy supply. The item of exceptional interest is the presence of the oxide of manganese.

	(11)	(12)	(13)
Silica (as SiO_2)	0·0	0·0	1·0
Iron (as Fe_2O_3)	trace	4·5	1·2
Baryta (as BaO)	1·38	28·36	2·23
Lime (as CaO)	2·60	141·64	15·54
Magnesia (as MgO)	trace	42·16	4·82
Soda (as Na_2O)	0·0	1080·76	114·02

Numbers (11), (12) and (13) are the analyses of numbers (26), (27) and (28) (see organic analyses, p. 31) from a bore

hole at Ilkeston. Number (11) is the original water, number (12) the abnormal water, number (13) from the pumping shaft. All these show the presence of soluble barium salts, a rare occurrence in a natural water in this country. A particular item to note is the extraordinary amount of soda which accounts for the very high chlorine in the organic analyses. The analyses were made by Mr J. White (*Analyst*, 1899, vol. XXIV, p. 67).

The following, No. (14), is the analysis of the Ilkley Spa water. (Analysis made by Mr B. A. Burrell, *Chem. News*, 1913, p. 295.)

	(14)
Ferric oxide (Fe_2O_3)	0·023
Calcium carbonate ($CaCO_3$)	1·157
Calcium nitrate ($Ca(NO_3)_2$)	0·020
Calcium silicate ($CaSiO_3$)	2·936
Calcium sulphate ($CaSO_4$)	0·742
Magnesium carbonate ($MgCO_3$)	2·033
Magnesium sulphate ($MgSO_4$)	1·557
Potassium carbonate (K_2CO_3)	0·221
Sodium carbonate (Na_2CO_3)	1·246
Sodium chloride (NaCl)	1·650
Lithium chloride (LiCl)	0·118

The height of the well from which this water is drawn is 660 ft. above sea level; the water issues from the middle or third grits of the Millstone Grit series and is clear and free from sediment.

	(15)
Lime	0·51
Magnesia	trace
Oxides of iron and alumina	0·34
Sulphates (as SO_3)	0·91
Sodium chloride	1·32

Number (15) is the analysis of the Glasgow City Water Supply (see organic analysis, number (21), p. 29) by F. W. Harris, Esq., City Analyst. As might be expected from the low total solid matter, nearly half of the mineral matter is sodium chloride. Note the complete absence of carbonates, which explains the absence of temporary hardness.

	(16)
Calcium sulphate ($CaSO_4$)	2·47
Magnesium sulphate ($MgSO_4$)	0·84
Magnesium chloride ($MgCl_2$)	0·73
Sodium chloride (NaCl)	0·70
Iron, Alumina and Silica	0·36

Number (16) is an analysis of the Manchester tap water derived from Lake Thirlmere. The total hardness is 2°. Note the absence of carbonate with the consequent absence of temporary hardness. The water is a pure, very soft siliceous water well suited for domestic and industrial uses.

	(17)
Calcium sulphate ($CaSO_4$)	32·18
Calcium carbonate ($CaCO_3$)	15·80
Magnesium sulphate ($MgSO_4$)	12·70
Magnesium carbonate ($MgCO_3$)	3·94
Potassium sulphate (K_2SO_4)	9·53
Potassium chloride (KCl)	2·68
Sodium chloride (NaCl)	12·99
Iron carbonate ($FeCO_3$)	0·82
Silica (SiO_2)	0·99

Number (17) is a typical hard water from the Keuper Marl at Burton containing a large temporary and permanent hardness. It is not suitable for domestic purposes, but is well suited for brewing Burton pale ales and also for malting purposes.

	(18)
Calcium carbonate ($CaCO_3$)	26·97
Magnesium carbonate ($MgCO_3$)	0·40
Sodium chloride (NaCl)	2·77
Sodium sulphate (Na_2SO_4)	1·06
Potassium silicate (K_2SiO_3)	0·92
Potassium sulphate (K_2SO_4)	0·54
Potassium carbonate (K_2CO_3)	0·37
Silica (SiO_2)	0·98

Number (18) is an analysis of a water from a London Chalk well. In this case all the hardness is temporary and there is no permanent. The water would require careful softening before being used for domestic or industrial purposes.

	(19)
Calcium sulphate ($CaSO_4$)	16·70
Calcium carbonate ($CaCO_3$)	28·37
Magnesium sulphate ($MgSO_4$)	15·57
Magnesium carbonate ($MgCO_3$)	7·82
Sodium sulphate (Na_2SO_4)	6·37
Sodium chloride (NaCl)	16·73
Potassium chloride (KCl)	4·08
Silica (SiO_2)	0·68

Number (19) is an analysis of a well water from the Edinburgh district. It is excessively hard (both temporary and permanent) and is not suitable for domestic and industrial purposes unless it is carefully softened.

The following analyses were kindly supplied by Mr West Knights. The numbers represent grains per gallon.

	(20)	(21)	(22)	(23)	(24)
Calcium carbonate	14·20	5·71	23·00	15·26	18·00
Magnesium carbonate ..	nil	2·56	—	—	—
Magnesium sulphate ..	9·00	nil	3·24	0·75	0·75
Sodium chloride	8·89	4·94	17·31	2·30	6·59
Sodium sulphate	19·91	8·34	—	0·40	—
Sodium carbonate	nil	3·45	—	—	—
Sodium nitrate	nil	nil	6·07	—	—
Calcium nitrate	—	—	—	—	5·85
Calcium sulphate	—	—	—	—	3·01
Not determined	—	—	4·38	0·29	—
Total solids	52·00	25·00	54·00	19·00	34·2

Number (20) is the analysis of the mineral constituents of a well in the Lower Greensand.

Number (21) is also from another well in the Lower Greensand.

Number (22) is from a shallow well in the Chalk.

Number (23) is from a deep well in the Chalk.

Number (24) is from a deep well near Newmarket.

By the courtesy of Dr A. C. Houston, the Director of Water Examinations, London Metropolitan Water Board, we are permitted to quote (on p. 39) some of the results of the chemical examination of the London water. The numbers represent parts per 100,000; and give the averages for the year 1911–12 of a considerable number of analyses.

The series of County Memoirs issued by the Geological Survey dealing with the water supplies derived from underground sources are of great importance to public health authorities. They contain a large amount of information relative to the geological nature of water-bearing strata, together with a considerable number of detailed analyses of waters both of the organic and the mineral constituents obtained from such strata. Each memoir has also a complete bibliography.

This table contains the average results of the chemical examination of the London water for the year 1911–12.

	Thames (*raw* water, 238 samples)	Lee (*raw* water, 238 samples)	New River (*raw* water, 238 samples)	New River (*filtered* water, 245 samples)	East London, Clapton (*filtered* water, 246 samples)	Chelsea (*filtered* water, 243 samples)
Ammoniacal nitrogen	0·0073	0·0106	0·0029	0·0017	0·0003	0·0002
Albuminoid nitrogen	0·0155	0·0156	0·0090	0·0033	0·0056	0·0059
Oxidised nitrogen	0·2600	0·3200	0·3100	0·2800	0·2400	0·2500
Chlorine	1·7600	2·1400	1·6200	1·7100	2·0800	1·7000
Oxygen absorbed from potassium permanganate in 3 hours at 80° F.	0·2062	0·2102	0·0920	0·0439	0·0719	0·0892
Total hardness	21·670	24·870	24·890	22·680	22·990	21·000
Permanent hardness	5·620	6·81	4·970	5·070	7·160	6·090

The table below contains the results of the mineral analyses of some of the London waters. The numbers are in parts per 100,000.

			Kent wells		Lee Valley wells	
	Thames (*raw* water, Nov. 15/09)	Lambeth (*filtered* water, Nov. 15/09)	(Deptford Garden well, Nov. 4/09)	(Darenth well, Jan. 11/10)	(Park well, Nov. 4/09)	(East Ham well, Jan. 14/10)
Calcium carbonate	23·00	21·64	24·20	23·23	17·57	8·66
Magnesium carbonate	nil	nil	nil	0·10	3·07	6·67
Sodium carbonate	nil	nil	nil	nil	nil	7·80
Calcium sulphate	3·28	3·47	9·20	nil	nil	nil
Magnesium sulphate	2·39	2·30	2·09	0·96	5·79	nil
Sodium sulphate	0·43	0·44	nil	0·67	3·90	11·06
Magnesium chloride	nil	nil	nil	nil	nil	nil
Chlorine as NaCl (+a little KCl)	2·82	2·93	3·43	2·24	3·28	12·10
Calcium nitrate	nil	nil	nil	nil	nil	nil
Magnesium nitrate	nil	nil	1·47	nil	nil	nil
Sodium nitrate	1·70	1·63	1·46	2·43	nil	nil
Sesquioxides of iron and alumina	0·06	0·01	0·12	nil	0·06	0·06
Silica	1·06	1·24	1·20	1·00	1·42	0·92
	34·74	33·66	43·17	30·63	35·09	47·27

SEWAGE.

In sampling a crude sewage, care must be taken to obtain a representative sample and it must be proportional to the rate of flow.

The person taking the sample should be instructed to take a portion of the inflow every hour; the contents of the various Winchester quarts collected during 24 hours should be mixed and a representative sample taken from the mixed portions. In order that the sample may represent the true sewage a sample should be taken on each of ten consecutive days.

Free and albuminoid ammonia in sewage and sewage effluents are estimated in accordance with the directions for water analysis (pp. 2, 3, 4) except that for the determination 5 c.c. of the sample are mixed with 500 c.c. of ammonia-free distilled water.

Nitrogen as nitrates and nitrites, total solid matter and chlorine are also determined as directed under water analysis (pp. 8—12).

Oxygen absorbed test in four hours at 80° F. The methods described on pp. 5—7 may be used, but 10 or 20 c.c. of the sewage (or sewage effluent) are taken and diluted to 250 c.c. with ammonia-free water before placing it in the bottle.

Oxygen absorbed in three minutes at 80° F. *Solutions required:*

Strong potassium permanganate solution. Weigh out 3·95 grams of pure potassium permanganate; dissolve in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 0·001 gram of available oxygen.

Sulphuric acid solution prepared as directed under water analysis (p. 6).

Oxalic acid solution. Weigh out 7·875 grams of crystallized oxalic acid; dissolve in distilled water and make up to 1000 c.c. 1 c.c. of this solution = 1 c.c. of the strong potassium permanganate solution.

For the estimation, measure out 10 c.c. of the well shaken sample into a conical flask after rinsing with concentrated sulphuric acid; add 240 c.c. of distilled water and 10 c.c. of

the sulphuric acid solution ; raise the temperature on the water bath to 80° F., and add 10 c.c. of the strong potassium permanganate solution (at 80° F.) from a pipette and maintain the temperature at 80° F. for three minutes.

At the end of three minutes, titrate the solution with the oxalic acid solution until the pink colour just disappears ; the difference between the number of c.c. of oxalic acid solution used in a blank experiment and in the sewage multiplied by 0·001 gives the amount of oxygen absorbed by 10 c.c. of the sewage in three minutes.

Example :

Sewage taken	10 c.c.
Strong potassium permanganate solution added	10 c.c.
Oxalic acid used for the blank	10·0 c.c.
Oxalic acid used for sewage	7·5 c.c.
	2·5

2·5 × 0·001 = 0·0025 part of oxygen absorbed by 10 c.c. of sewage = 25·0 parts of oxygen absorbed per 100,000.

Estimation of suspended matter. 250 c.c. of the well shaken sample are filtered through a dried and weighed filter paper, the suspended matter on the paper is washed with water, dried in the water oven, and weighed, the weight of the filter paper subtracted from the total weight gives the weight of suspended matter in 250 c.c. of the sample. The filter paper containing the suspended matter is then placed in a platinum dish and incinerated ; from the weight of the ash is subtracted the weight of the filter paper ash, giving the weight of mineral matter in suspension.

Example :

Weight of filter paper, weighing bottle and suspended matter	20·133
Weight of filter paper and weighing bottle ..	19·999
Weight of suspended matter	·134

0·134 × 4 × 100 = 53·6 parts of suspended matter per 100,000.

Weight of dish, mineral matter and filter paper ash	48·4470
Weight of dish	48·4440
Weight of mineral matter and filter paper ash ..	·0030
Weight of filter paper ash	·0005
Weight of mineral suspended matter	·0025

$0{\cdot}0025 \times 4 \times 100 = 1{\cdot}0$ part of mineral suspended matter per 100,000.

Estimation of total organic nitrogen. *Solutions required :*

Phenol sulphuric acid solution.

Weigh out 200 grams of phosphoric anhydride.

40 grams of phenol.

Measure out 1000 c.c. of concentrated sulphuric acid.

Dissolve the phosphoric anhydride in part of the sulphuric acid and the phenol in the remainder, cool the two solutions and mix them.

The processes used for the estimation is the Kjeldahl-Jodlbaur method, in which 250–500 c.c. of the crude sewage are evaporated with 25 c.c. of the phenol-sulphuric acid in a 700 c.c. Kjeldahl flask; three grams of zinc dust and one gram of mercury are then added and the mixture allowed to stand for a short time, in order to allow the nitrates to be reduced. After the nitrates have been reduced, the flask is heated until the contents are colourless, the flame being allowed to play directly on the flask. When the contents of the flask are colourless, the flame is removed and the flask allowed to cool; when quite cold the contents are washed out into a 1000 c.c. boiling flask with distilled water and made alkaline with caustic soda solution; 20 c.c. of a 10 per cent. potassium sulphide solution are added, to precipitate the mercury; a few pieces of pipeclay are placed in the flask, which is then connected to a condenser and the ammonia distilled off and collected in an excess of a normal solution of sulphuric acid, placed in the receiver. As soon as all the ammonia has been distilled over, the excess of acid in the receiver is titrated with normal caustic soda solution; the number of c.c. of acid taken less the number of c.c. of soda solution used multiplied by 0·014 gives the total nitrogen in the volume of sewage taken.

A blank experiment should be performed with the materials used in order to determine the amount of nitrogen present in them, and the nitrogen so obtained should be subtracted from the total nitrogen.

Example:

500 c.c. of the sample taken for the estimation gave the following results:

Normal sulphuric acid placed in receiver ..	10·0 c.c.
Normal soda solution used to neutralize excess of acid	6·5 c.c.
	3·5 c.c.
Less acid used in the blank experiment	·3 c.c.
Acid neutralized by the ammonia from the sewage	3·2 c.c.

$3{\cdot}2 \times 0{\cdot}014 \times 200 = 8{\cdot}96$ parts of total nitrogen per 100,000.

Strength of sewage. The strength of a sewage can be calculated with a fair degree of accuracy by McGowan's formula (*Royal Commission on Sewage Disposal*, 5th Report, p. 16, also appendix IV to this Report), which is:—the organic nitrogen plus the ammoniacal nitrogen multiplied by 4·5 plus the oxygen absorbed in four hours multiplied by 6·5 gives a figure upon which the strength is based (or Ammon. N. + Org. N.) $\times$ 4·5 + (oxygen absorbed in four hours $\times$ 6·5). For example a sewage may be graded as follows:

Weak is	.. under 60
Medium is	.. 50–100
Average is	.. 100–175
Strong is	.. above 175

A rough guide to the strength of a crude sewage may also be obtained from the figure for the oxygen absorbed test in four hours at 80° F.: for example:

Weak	7–8	parts per 100,000
Average	10–12	,, ,,
Strong	17–25	,, ,,

Typical Analyses of a crude sewage in parts per 100,000.

Free ammonia	7·31	4·43	5·0	10·61	16·58
Albuminoid ammonia ..	1·52	1·07	1·8	1·42	2·21
Oxygen absorbed in three minutes at 80° F.	—	3·51	12·8	5·30	9·50
Oxygen absorbed in four hours at 80° F.	17·81	13·99	24·0	19·81	21·19
Total solid matter	—	—	178·9	—	—
Chlorine	10·30	9·88	15·2	16·28	17·12
Nitrogen as nitrate	nil	nil	nil	nil	nil
Suspended matter	17·6	46·5	86·0	49·50	62·1

A typical Analysis of a crude sewage containing trade waste.

	parts per 100,000
Free ammonia	2·3
Albuminoid ammonia	3·7
Oxygen absorbed in three minutes at 80° F.	50·7
Oxygen absorbed in four hours at 80° F. ..	69·2
Total solid matter	216·0
Chlorine	48·0
Nitrogen as nitrate	nil
Suspended matter	40·0

Analyses of crude sewage obtained from the same source on seven consecutive days showing variations in the composition.

Free ammonia	3·5	3·5	5·4	3·2	4·4	4·0	4·0
Albuminoid ammonia ..	1·4	1·8	1·8	1·8	2·4	1·9	1·6
Oxygen absorbed in three mins. at 80° F. ..	4·7	8·4	9·7	6·4	14·4	8·8	7·2
Oxygen absorbed in four hours at 80° F. ..	14·9	20·6	26·6	25·5	29·5	21·6	21·5
Total solid matter	120·0	144·0	184·0	140·0	172·0	136·0	110·0
Chlorine ..	10·0	14·4	16·8	12·7	18·8	13·2	12·4
Nitrogen as nitrate	nil	nil	nil	nil	nil	nil	nil
Suspended matter	40·0	52·0	90·0	48·0	52·0	36·0	80·0
Strength ..	average	average	strong	strong	strong	average	average

SEWAGE EFFLUENTS.

It is important from a Public Health point of view that a stream should not be polluted with a sewage or sewage effluent which might be harmful to the animal life contained therein, or which might produce a nuisance or cause disease to be carried to the towns and villages near which the stream passes on its way to the sea.

An enormous amount of work has been performed on sewage and their effluents by the officers of the Royal Commission on Sewage Disposal and the results of this work are published in the various reports of the Commission. A large number of analyses of all kinds of sewages and sewage effluents are contained in these reports.

In the *8th Report*, vol. I of the Commission a standard is formulated, with which it is recommended that all effluents should comply before being poured into a stream. In the suggested standard a normal sewage effluent should not contain more than three grams per 100,000 of suspended matter and should not take up more than two grams of dissolved oxygen per 100,000 at 65° F. in five days: but the Report also discusses cases where, owing to the relatively small volume of the river, which is the receiver of sewage effluents, a more stringent standard is necessary: and, on the other hand, conditions of dilution which indicate that a relaxation of the normal standard may be allowed. Methods of determining the suspended matter and the dissolved oxygen absorbed are also given. (*8th Report*, vol. II, appendix.)

Determination of suspended matter.

Preparation of the asbestos; a considerable quantity of asbestos floss is well washed with water, digested with hydrochloric acid, and, afterwards, thoroughly washed with water until all traces of hydrochloric acid are removed.

The process. Sufficient of the washed asbestos is placed in a Gooch crucible to form a layer 0·2 cm. deep; the crucible is fixed to a pressure filter flask and the pump turned on; the asbestos is spread evenly over the bottom of the crucible and well rammed down; a gentle stream of water is run through

the crucible all the time. After the asbestos has been well packed in the crucible, 250 c.c. of tap water are run through, to remove all loose particles, and finally a few c.c. of distilled water; the crucible is then dried in the air oven at 105° C., ignited and weighed.

The amount of effluent taken for the determination of the suspended matter varies considerably, but the average is 200 c.c. A small quantity of this 200 c.c. is used to moisten the asbestos; the pump is then turned on very gently and as much as possible of the liquid is run through under this gentle pressure; the pressure is increased towards the end until the 200 c.c. have passed through the crucible; the contents of the crucible are now washed with distilled water; dried in the air oven at 105° C. and weighed regularly till the weight is constant; the increase in weight of the crucible represents the total suspended matter in 200 c.c. of the effluent.

The crucible is now ignited at a low red heat, allowed to cool and reweighed; the loss in weight is due to volatile matter, and this subtracted from the total gives the mineral matter. The determination should always be made in duplicate.

Example: 200 c.c. of a sewage effluent were taken for the estimation; the increase in weight of the crucible was 0·0812 gram. The total suspended matter therefore was

$$0{\cdot}0812 \times 500 = 40{\cdot}6 \text{ parts per } 100{,}000.$$

After ignition the crucible had lost in weight 0·052 gram, the volatile matter was $0{\cdot}052 \times 500 = 26{\cdot}0$ parts per 100,000, $40{\cdot}6 - 26{\cdot}0 = 14{\cdot}6$ parts per 100,000 of mineral matter.

Determination of the dissolved oxygen.

Winkler's method (*Ber.* 1888, XXI, p. 2843) as modified by Rideal and Stewart (*Analyst*, 1901, XXVI, p. 141); see also *8th Report*, vol. II, Appendix of the Royal Commission on Sewage Disposal, p. 97. The principle on which it depends is the oxidation in an alkaline liquid of manganous oxide to a higher oxide of manganese, the subsequent liberation of iodine from potassium iodide by this in the acidified solution, and the titration of the liberated iodine by sodium thiosulphate.

Solutions required :

Concentrated sulphuric acid.

Concentrated hydrochloric acid (free from chlorine).

N/8 potassium permanganate solution (3·94 grams $KMnO_4$ per litre).

A 2 per cent. solution of potassium oxalate ($K_2C_2O_4 + H_2O$ —crystals).

A 33 per cent. solution of manganous chloride ($MnCl_2 + 4H_2O$—crystals).

A solution containing 70 grams of caustic potash and 10 grams of potassium iodide per 100 c.c.

A solution of N/20 sodium thiosulphate (12·4 grams per 1000 c.c.).

The estimation is made in bottles of 340 to 360 c.c. capacity, with well-fitting ground glass stoppers; in cold weather the bottles are warmed to 18° C. (65° F.).

Shake the bottle containing the effluent thoroughly well in order to bring the dissolved oxygen content to something near that of the diluting water; measure out into a flask of ample size one volume of the effluent and four volumes of tap water at 18° C.; mix these well and fill four of the bottles completely with the mixture; the bottles, which must be filled quietly and quickly, are then left unstoppered to remove any air bubbles; at the end of five minutes, the stoppers are replaced in the bottles and two of the bottles are placed in the incubator at 18° C. (65° F.) for five days, the other two being used for the determination at once.

To each bottle add 0·9 c.c. of the concentrated sulphuric acid; then sufficient of the potassium permanganate solution to leave a slight pink colour (1 to 2 c.c. is usually enough); mix the contents of the bottles and allow them to stand for 20 minutes with the stoppers in the bottles; this is to oxidize any nitrites to nitrates; at the end of 20 minutes, about 1 c.c. of the potassium oxalate solution is added to each bottle in order to destroy the excess of potassium permanganate, the bottles are at once restoppered and the contents well mixed.

As soon as the contents are colourless, the stoppers are removed and 1 c.c. of the manganous chloride solution is run

into each bottle from a pipette, the tip of the pipette being at the bottom of the bottle; immediately afterwards 4 c.c. of the caustic potash-potassium iodide solution are added again at the bottom of the bottle; the stoppers are inserted and the bottles turned over once or twice, and after a few minutes again turned over; the bottles are then allowed to stand for a few minutes to allow the hydroxides of manganese to settle. After standing, 5 c.c. of hydrochloric acid are run into each bottle from a pipette again at the bottom; the bottles are restoppered and kept in a dark place for ten minutes, being rotated occasionally; after standing for ten minutes, 20 c.c. of the contents are pipetted out of each bottle and rejected; the remainder of the contents in the bottle is titrated with the sodium thiosulphate solution till colourless; using a clear starch solution as an indicator towards the end. The oxygen equivalent of the sodium thiosulphate being known, the amount of the dissolved oxygen is easily calculated. The mean of the two determinations is taken.

After five days' incubation, the oxygen remaining in solution in the other two bottles is determined in the same way; the difference between the two determinations represents the dissolved oxygen absorbed by the effluent in the interval.

The dissolved oxygen is calculated as follows:

N = the number of c.c. of the sodium thiosulphate solution used.

S = the strength of the sodium thiosulphate solution in grams of oxygen per c.c.

v = the volume of the bottle in c.c.

$$\therefore \text{ the dissolved oxygen in parts per } 100{,}000 = \frac{100{,}000 \times N \times S}{v}.$$

Another method employed by the Royal Commission on Sewage Disposal (*5th Report*, appendix 6, pp. 221–226) for estimating the amount of oxygen dissolved in water gives excellent results. It is described by Letts and Blake (*Proc. Roy. Dublin Soc.* vol. IX. (N.S.), pt. 4, No. 33) and has been used by the New York Metropolitan Sewage Commission (*Report*, 1912, p. 303).

Reagents required: standard ferrous sulphate is prepared by

dissolving 144 grams of the pure crystallized salt in distilled water; add 15 c.c. of strong sulphuric acid: dilute the whole to three litres.

Standard sodium carbonate. Dissolve 100 grams of the crystals of pure sodium carbonate in one litre of water.

Standard sulphuric acid. Dissolve one part of concentrated sulphuric acid in an equal volume of distilled water.

Standard potassium permanganate. Dissolve 25·4 grams of pure potassium permanganate in distilled water and dilute to 4·5 litres with distilled water.

Method of testing. The volume of the separating flask is first measured by filling it with distilled water; replace the stopper and then run the water into a measuring cylinder. The flask is afterwards filled with the water to be analysed. Six c.c. of the standard ferrous sulphate solution and 4 c.c. of the sodium carbonate solution are added by means of a pipette and delivered near the bottom of the flask; replace the stopper and shake the contents. The dissolved oxygen in the presence of the sodium carbonate oxidizes a portion of the ferrous sulphate which is precipitated. Allow to stand five minutes: then invert the flask, and introduce 10 c.c. of the standard sulphuric acid through the small bulb of the flask, by means of the stopcock. The acid is allowed to mix thoroughly with the other contents of the flask, and it is allowed to stand until the mixture is almost colourless. The contents of the flask are then emptied into a dish and titrated in the usual way with the standard potassium permanganate solution. A blank sample is then analysed. In this case the flask is filled with the water as before; 10 c.c. of the standard sulphuric acid are added and the whole shaken well; add 6 c.c. of the standard ferrous sulphate and shake again; empty the flask into a dish and titrate with the standard potassium permanganate solution. The addition of the acid in this part of the experiment prevents the dissolved oxygen from acting upon the ferrous sulphate.

Subtract the result of the titration of the first sample from that of the second and the amount of the ferrous sulphate oxidized by the oxygen dissolved in the water is thus obtained. The results should be stated in terms of the number of c.c. of

dissolved oxygen in one litre of water. Suppose for example 1 c.c. of the potassium permanganate solution is equal to 1·009 c.c. of oxygen at 0° C. and 760 mm. pressure, the amount of the difference between the two titrations is multiplied by 1·009 and by 1000 and divided by the volume content, in c.c.'s, of the analysing flask. If it is required to determine the percentage of the saturation of the sample of water, it can be done by calculating from a diagram prepared by the Commissioners for instances of sea and land waters contained on p. 58 of Appendix 6 of the *5th Report of the Royal Commission on Sewage Disposal*. This diagram gives the saturation figures at different temperatures for sea water and distilled water. (See Addenda of this book, p. 214 for table of solubilities of oxygen and nitrogen in distilled water and sea water.)

Typical analyses of various sewage effluents (in parts per 100,000).

Ammoniacal nitrogen	0·02	0·03	0·26	1·40	0·26	1·66	2·12
Albuminoid nitrogen ..	0·10	0·07	0·27	0·24	0·16	0·27	0·18
Nitrous nitrogen ..	trace	trace	0·02	0·04	nil	0·03	0·02
Nitric nitrogen ..	1·92	3·10	2·28	1·83	0·45	0·61	0·55
Oxygen absorbed in four hours at 80° F.	0·87	0·87	3·41	2·33	1·90	1·91	1·54
Solids in suspension ..	2·00	0·70	8·10	4·40	3·20	2·80	0·90
Oxygen in solution ..	nil	—	—	—	—	—	—
Dissolved oxygen taken up in five days at 65° F.	0·34	0·39	3·10	3·44	3·12	3·77	0·68
Remarks	earthy smell; bright; clear; small amount of fine brown solids	bright; no smell; trace of suspended matter	turbid; considerable brown suspended matter	brownish; opalescent; strong fishy smell	turbid; earthy smell	brownish; opalescent; strong fishy smell	brownish slightly opalescent; fishy smell

See also the *4th Report of the Royal Commission on Sewage Disposal* (1904), vol. IV, parts 2 and 5, which describe methods of chemical analysis as applied to sewage and sewage effluents, and also contain tables of analyses.

RIVER WATER AND SEWAGE.

The following tables (pp. 51—56) contain the results of the determinations of the amount of oxygen dissolved in, and taken up by, a river, a sewage effluent and mixtures of the effluent with a river during the summer and winter months of 1913. They

Tables containing the results of the determinations of the amount of oxygen dissolved and absorbed by the effluent and the river at various times.

Date 1913	Hour of collecting	Location of the sample	Temperature of the water C.	Barometric pressure in mm.	Dilution	Feet below the surface	Total suspended solids in grams per 100,000	Oxygen dissolved in c.c. per litre at 0° C. and 760 mm.		Oxygen absorbed in five days in grams per 100,000	Putrefaction on incubation at 18° C.
								At the commencement	After five days' incubation at 18° C.		
May 29	1 p.m.	River 150 feet above the effluent outfall	20	760	—	1·5	—	7·183	6·208	0·142	—
,,	,,	Sewage effluent	20	,,	—	—	3·2	0·807	nil	9·903	H_2S present
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	20	,,	1 to 15	1·5	—	5·557	2·822	0·392	—
,,	,,	River ¼ mile below the effluent outfall	20	,,	—	1·5	—	7·057	5·180	0·338	—
June 12	10.30 a.m.	River 150 feet above the effluent outfall	15	764	—	1·5	—	7·776	7·479	0·041	—
,,	,,	Sewage effluent	15	,,	—	—	2·9	0·230	nil	6·451	H_2S present
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	15	,,	1 to 16	1·5	—	7·559	3·465	0·586	—
,,	,,	River ¼ mile below the effluent outfall	15	,,	—	1·5	—	8·651	4·934	0·561	—
June 13	10 a.m.	River 150 feet above the effluent outfall	16	767	—	1·5	—	10·086	9·268	0·117	—
,,	,,	Sewage effluent	16	,,	—	—	2·8	2·727	nil	4·3	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	16	,,	1 to 16	1·5	—	9·796	7·912	0·270	—
,,	,,	River ¼ mile below the effluent outfall	16	,,	—	1·5	—	10·057	7·437	0·375	—

The River Cam and the Sewage Effluent of Cambridge (contd.).

Date 1913	Hour of collecting	Location of the sample	Temperature of the water ° C.	Barometric pressure in mm.	Dilution	Feet below the surface	Total suspended solids in grams per 100,000	Oxygen dissolved in c.c. per litre at 0° C. and 760 mm. At the commencement	Oxygen dissolved in c.c. per litre at 0° C. and 760 mm. After five days' incubation at 18° C.	Oxygen absorbed in five days in grams per 100,000	Putrefaction on incubation at 18° C.
July 10	10 a.m.	River 150 feet above the effluent outfall	16	758	—	1·5	—	7·793	7·496	0·042	—
,,	,,	Sewage effluent	—	Sample Bottle lost in sewer				—	—	—	—
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	16	758	1 to 15	1·5	—	7·311	5·849	0·209	—
,,	,,	River ¼ mile below the effluent outfall	16	,,	—	1·5	—	8·542	7·487	0·151	—
July 17	10 a.m.	River 150 feet above the effluent outfall	17	762	—	1·5	—	5·693	5·397	0·041	—
,,	,,	Sewage effluent	17	,,	—	—	3·0	1·098	nil	7·066	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	17	,,	1 to 15	1·5	—	5·556	2·942	0·377	—
,,	,,	River ¼ mile below the effluent outfall	17	,,	—	1·5	—	6·818	4·428	0·342	—
July 24	1 p.m.	River 150 feet above the effluent outfall	15	766	—	1·5	—	8·364	7·474	0·127	—
,,	,	Sewage effluent	15	,,	—	—	3·3	1·386	nil	4·547	H_2S present
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	15	,,	1 to 17	1·5	—	8·188	spoilt 7·018	spoilt 0·167	—
,,	,,	River ¼ mile below the effluent outfall	15	,,	—	1·5	—	8·949	6·553	0·342	—

Date 1913	Hour of collecting	Location of the sample	Temperature of the water °C.	Barometric pressure in mm.	Dilution	Feet below the surface	Total suspended solids in grams per 100,000	Oxygen dissolved in c.c. per litre at 0° C. and 760 mm.		Oxygen absorbed in five days in grams per 100,000	Putrefaction on incubation at 18° C.
								At the commencement	After five days' incubation at 18° C.		
July 31	10.30 a.m.	River 150 feet above the effluent outfall	17	762	—	1·5	—	7·479	6·886	0·085	—
,,	,,	Sewage effluent	17	,,	—	—	1·7	1·959	nil	2·919	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	17	,,	1 to 12	1·5	—	7·019	5·557	0·209	—
,,	,,	River ¼ mile below the effluent outfall	17	,,	—	1·5	—	8·120	6·792	0·190	—
Aug. 7	12 m.	River 150 feet above the effluent outfall	16	762	—	3	—	8·661	8·067	0·085	—
,,	,,	Sewage effluent	16	,,	—	—	6·2	2·251	nil	5·220	H_2S present
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	16	,,	1 to 14	3	—	7·311	0·876	0·922	—
,,	,,	River ¼ mile below the effluent outfall	16	,,	—	3	—	8·942	6·818	0·304	—
Aug. 14	10.30 a.m.	River 150 feet above the effluent outfall	17	763	—	3	—	7·479	6·589	0·127	—
,,	,,	Sewage effluent	17	,,	—	—	2·5	1·959	nil	0·617	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	17	,,	1 to 17	3	—	6·727	4·972	0·251	—
,,	,,	River ¼ mile below the effluent outfall	17	,,	—	3	—	8·385	7·323	0·152	—

The River Cam and the Sewage Effluent of Cambridge (contd.).

Date 1913	Hour of collecting	Location of the sample	Temperature of the water ° C.	Barometric pressure in mm.	Dilution	Feet below the surface	Total suspended solids in grams per 100,000	Oxygen dissolved in c.c. per litre at 0° C. and 760 mm.		Oxygen absorbed in five days in grams per 100,000	Putrefaction on incubation at 18° C.
								At the commencement	After five days' incubation at 18° C.		
Oct. 10	12 m.	River 150 feet above the effluent outfall	12	764	—	3	—	7·179	5·101	0·297	—
,,	,,	Sewage effluent	12	,,	—	—	10·0	1·389	nil	1·804	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	12	,,	1 to 17	3	—	6·142	3·802	0·335	—
,,	,,	River ¼ mile below the effluent outfall	12	,,	—	3	—	7·379	5·743	0·234	—
Oct. 18	12 m.	River 150 feet above the effluent outfall	12	764	—	3	—	7·852	5·991	0·266	—
,,	,,	Sewage effluent	12	,,	—	—	4·0	3·799	nil	5·680	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	12	,,	1 to 17	3	—	8·515	5·264	0·466	—
,,	,,	River ¼ mile below the effluent outfall	12	,,	—	3	—	8·306	7·106	0·172	—
Oct. 29	10 a.m.	River 150 feet above the effluent outfall	12	744	—	3	—	6·292	3·649	0·378	—
,,	,,	Sewage effluent	12	,,	—	—	1·0	2·824	nil	3·001	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	12	,,	1 to 17	3	—	5·263	1·769	0·500	—
,,	,,	River ¼ mile below the effluent outfall	12	,,	—	3	—	6·813	4·042	0·397	—

Date 1913	Hour of collecting	Location of the sample	Temperature of the water °C.	Barometric pressure in mm.	Dilution	Feet below the surface	Total suspended solids in grams per 100,000	Oxygen dissolved in c.c. per litre at 0° C. and 760 mm.		Oxygen absorbed in five days in grams per 100,000	Putrefaction on incubation at 18° C.
								At the commencement	After five days' incubation at 18° C.		
Nov. 5	12 m.	River 150 feet above the effluent outfall	10	752	—	3	—	6·300	4·539	0·252	—
,,	,,	Sewage effluent	10	,,	—	—	6·1	2·238	nil	3·045	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	10	,,	1 to 17	3	—	4·468	1·184	0·470	—
,,	,,	River ¼ mile below the effluent outfall	10	,,	—	3	—	7·046	4·315	0·391	—
Nov. 12	11 a.m.	River 150 feet above the effluent outfall	9	744	—	3	—	6·913	4·231	0·384	—
,,	,,	Sewage effluent	9	,,	—	—	1·3	1·961	nil	2·484	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	9	,,	1 to 17	3	—	5·559	nil	0·796	—
,,	,,	River ¼ mile below the effluent outfall	9	,,	—	3	—	6·496	2·371	0·591	—
Nov. 19	10.30 a.m.	River 150 feet above the effluent outfall	8	771	—	3	—	7·776	5·995	0·255	—
,,	,,	Sewage effluent	8	,,	—	—	2·6	2·824	nil	2·752	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	8	,,	1 to 15	3	—	6·705	1·022	0·814	—
,,	,,	River ¼ mile below the effluent outfall	8	,,	—	3	—	8·176	5·723	0·351	—

The River Cam and the Sewage Effluent of Cambridge (contd.).

Date 1913	Hour of collecting	Location of the sample	Temperature of the water ° C.	Barometric pressure in mm.	Dilution	Feet below the surface	Total suspended solids in grams per 100,000	Oxygen dissolved in c.c. per litre at 0° C. and 760 mm.		Oxygen absorbed in five days in grams per 100,000	Putrefaction on incubation at 18° C.
								At the commencement	After five days' incubation at 18° C.		
Nov. 26	11 a.m.	River 150 feet above the effluent outfall	8	769	—	3	—	7·479	5·406	0·297	—
,,	,,	Sewage effluent	8	,,	—	—	0·292	3·112	nil	2·592	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	8	,,	1 to 20	3	—	7·842	3·002	0·693	—
,,	,,	River ¼ mile below the effluent outfall	8	,,	—	3	—	8·722	6·248	0·354	—
Dec. 3	11.30 a.m.	River 150 feet above the effluent outfall	9	755	—	3	—	8·073	6·297	0·254	—
,,	,,	Sewage effluent	9	,,	—	—	7·0	3·112	nil	2·873	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	9	,,	1 to 17	3	—	7·544	4·422	0·447	—
,,	,,	River ¼ mile below the effluent outfall	9	,,	—	—	—	8·722	5·702	0·432	—
Dec. 8	11.30 a.m.	River 150 feet above the effluent outfall	5	767	—	3	—	8·373	5·995	0·340	—
,,	,,	Sewage effluent	5	,,	—	—	2·1	2·531	nil	2·334	nil
,,	,,	Mixture of effluent and river water 50 feet below effluent outfall	5	,,	1 to 15	3	—	7·550	3·579	0·596	—
,,	,,	River ¼ mile below the effluent outfall	5	,,	—	3	9·4	8·293	4·359	0·563	—

are extracted from an investigation by Purvis and Black on the oxygen content of the river Cam before and after receiving the Cambridge sewage effluent (*Proc. Camb. Phil. Soc.* vol. XVII, pt V, p. 353). They are typical results on the basis of the standard of the amount of dissolved oxygen which is consumed in five days at 18° C. suggested by the *8th Report of the Royal Commission on Sewage Disposal* (1912), vol. I.

The more important facts which arise from a comparison of the above analyses are the following: (*a*) The solids in suspension in the effluent were, on several occasions in the summer months, above the standard of three grams per 100,000; and on these occasions an offensive smell was noticed after five days' incubation. On four occasions during the winter months the suspended solids were also above the standard; but, on the other hand, there was no smell after five days' incubation. On examining these solids microscopically it was found that they chiefly consisted of zoogloea masses of a filamentous bacillus which had developed and grown on the inside of the drain pipes and inspection chambers carrying the effluent to the river. They were not fæcal substances which produced an unpleasant smell like those noticed in the summer. (*b*) The amount of oxygen absorbed by the river above the outfall was always above the standard of a clean river water (0·2 gram) in the winter months from October to December inclusive, whereas in the summer months, from May to August inclusive, it was always below the standard. It cannot be said, therefore, that the river itself always satisfies the standard of the Royal Commission. (*c*) Only on two occasions (August 14 and October 10) was the amount of oxygen taken up by the effluent below the standard of the Commissioners. (*d*) On the other hand, the purification which takes place when the effluent mixes with the river water is fairly rapid; and, although the mixture of the effluent and river at 50 feet below the outfall gives a figure which is sometimes above and sometimes below the standard of 0·4 gram of oxygen absorbed in five days at 18° C., yet the river $\frac{1}{4}$ mile down as regards its cleanliness compares fairly well with the standards of a diluting water suggested by the Commissioners. For example, five of the analyses would grade the

river as " clean," ten as " fairly clean " and three as " doubtful " at ¼ mile below the effluent outfall.

It will be noticed that the differences in the summer and winter months are very striking.

Three factors at least may be suggested to explain these differences and these are : (1) the varying amount of the rainfall, with which is connected a considerable amount of pollution derived mainly from road washings and the surface water from agricultural land, (2) the number of hours of sunshine, with which is connected the purifying power of the oxygen given out by aquatic plants under its influence, and (3) the changes in the temperature of the water (and therefore closely allied to the second factor), with which is connected the varying amounts of oxygen dissolved from the air at different temperatures. As the rainfall increases the amount of oxygen absorbed by the river in five days increases, and this was particularly noticeable in the rainy season in October. In the summer months, the number of hours of sunshine was greater than in the winter months, and the amount of oxygen in the river was increased as a consequence of the response to this influence by the aquatic plants. In the winter months, on the other hand, from November 12 to December 8, there was a distinct rise in the amount of oxygen dissolved, and this was explained by the decrease in the rainfall, and a fall in the temperature of the water, so that there was a kind of balance. In the summer months, the decreased solubility of the oxygen of the air caused by the higher temperature was more than compensated by the increased discharge of the oxygen from the aquatic plants ; and the increased solubility of the oxygen of the air caused by the lower winter temperature partly compensated for the disappearance of the oxygen discharged by the aquatic plants.

At the same time, as Letts has indicated (6th appendix (1908), *5th Report of the Royal Commission on Sewage Disposal*) in his experiments with the sea weed *Ulva latissima*, it is most likely that aquatic plants generally may be of considerable importance in the purification of a polluted stream. They are able to absorb some of the ammonia salts and nitrates, in

Average Chemical (in parts per 100,000) and Bacterial (in 1 c.c.) Analyses of the Cam Water above and below the Effluent Outfall.

	100 feet above Outfall	Crude Effluent	8 feet from Outfall	¼ mile below Outfall	½ mile below Outfall	¾ mile below Outfall	1 mile below Outfall	1½ miles below Outfall	2 miles below Outfall	2½ miles below Outfall	3 miles below Outfall	4 miles below Outfall
Free and saline ammonia	·068	—	·379	·112	·0846	·102	·074	·0712	·0862	—	—	—
Albuminoid ammonia ..	·0336	—	·0846	·032	·0286	·034	·035	·0275	·0412	—	—	—
Oxygen absorbed in 4 hours at 80° F. ..	·3143	—	·678	·2039	·2035	·254	·222	·216	·2213	—	—	—
Nitrogen as nitrates ..	·55	—	·55	·65	·583	·65	·575	·575	·575	—	—	—
Nitrogen as nitrites ..	Only traces were found, but there was a tendency to an increase down the river.											
Chlorine	3·06	—	4·483	3·4	3·4	3·5	3·35	3·35	3·35	—	—	—
B. coli found in 1 c.c. ..	661	102,422	27,522	—	6,670	—	14,443	8,540	6,377	2,349	1,525	30

addition to their ability to give off oxygen under the influence of sunshine.

Besides the above estimations of the amount of oxygen dissolved in, and taken up by, a river, the table on p. 59 contains the results of a series of chemical and bacterial analyses of the river Cam in an investigation by Purvis and Rayner (*Jour. Roy. San. Inst.* 1913, vol. XXXIV (X), p. 478). They show the chemical and bacterial (as regards *Bacillus coli*) variations at different parts beginning at 100 feet above the effluent outfall and extending four miles below it. There is some further pollution at two miles below the outfall. The samples were taken from 18 inches below the surface of the river and in midstream, and were obtained between 6 a.m. and 8 a.m. The numbers are the mean of three analyses on two days in May and one day in June, 1912.

It is clear from the table on p. 59 that the river is polluted at a place two miles below the effluent. The bacterial analysis shows marked pollution at about three quarters of a mile down, and the chemical figures are higher than at half a mile; and, even at two miles, the two ammonia figures are higher than at 100 feet above the effluent.

It is of some importance, however, to note that the figure for the absorbed oxygen goes down very rapidly, so that at a quarter of a mile it is below that at 100 feet above the outfall; it increases at three quarters of a mile and again rapidly decreases, so that at two miles it is again lower than at 100 feet above the outfall. There is, therefore, definite proof that the dissolved oxygen in the stream comes at once into action, and thereby decreases the amount of easily oxidizable substances in the effluent. The results are quite comparable with those of the oxygen absorbed figures described before, in Purvis and Black's observations.

The river therefore purifies itself chemically from the contaminating effluent quite satisfactorily; for between half a mile and three quarters of a mile, the albuminoid ammonia and the oxygen absorbed figures are lower than at 100 feet above the effluent outfall.

The dangerous pollution, as indicated by *B. coli*, coming

from the discharge of the sewage effluent into the stream, is still well marked at between three and four miles below the effluent outfall. It will be observed, however, that there is a definite amount of purification in this respect at four miles below, as compared with that at 100 feet above, the outfall. If the river were to receive no further pollution after that which is poured into it from the effluent outfall, it is, perhaps, probable that at about three miles below the outfall the water would be comparatively free from *Bacillus coli*.

See also the *8th Report of the Royal Commission on Sewage Disposal*, vol. II, appendix, on standards and tests for sewage and sewage effluents discharging into rivers and streams.

SEA-WATER AND SEWAGE.

An analysis of sea-water from the British Channel is given below. There are variations in such a sample and one taken from the Irish Sea or the North Sea.

	parts per 1000
Sodium chloride	28·00
Magnesium sulphate	2·30
Magnesium chloride	3·60
Magnesium bromide	0·03
Calcium sulphate	1·30
Calcium carbonate	0·03
Potassium chloride	0·74
Iodides	traces

Thorp and Morton (*Jahresb. für Chemie*, 1870, p. 1380) state that the Irish Sea contained 0·002 part of magnesium nitrate per 1000 of the water. This represents less than 0·04 part of nitrogen per 100,000 parts of water. It is difficult to realize that this represents the final product of the nitrification of the immense amount of organic nitrogenous compounds including nitrates which must have been poured in the sea, and which is continually being poured in from rivers of all kinds and all sizes. The matter is of considerable importance when sewage and sewage effluents are discharged into the sea or rivers; for it is desirable to know the nature and extent of the chemical, as well as the bacterial purification together with the length of time such purification may take.

Sea-water usually contains little or no free and saline ammonia, and very small amounts of albuminoid ammonia, whereas

nitrogen as nitrates and nitrites has rarely been found, and then in very small quantities. The possibilities of pollution of tidal estuaries, and indeed of the sea itself by the sewage poured into it from towns situated on the coast, is a serious matter. The Royal Commission on Sewage Disposal has collected a large amount of evidence from places where sewage has been deposited in the sea and tidal estuaries. Of great importance is the evidence in vol. VII of the Commissioners' Report given by Dr W. E. Adeney and Professor Letts. These investigators in a series of laboratory experiments mixed sea-water with 1 per cent. of sewage, and from time to time they estimated the two ammonias, and the nitrites and nitrates. They concluded from their experiments that nitrifying organisms can exist in such mixtures, although it was stated to be very remarkable that a large proportion of free ammonia remained unnitrified even after 11 months' inoculation. And, further, in Adeney and Letts' later experiments they found that the nitrates present at the beginning of the incubation disappeared after an interval of 21 months.

In an investigation by Purvis and Coleman[1] on "The influence of the saline constituents of sea-water on the decomposition of sewage," they give the results of the estimation of the two ammonias and of the nitrates and nitrites when sewage was incubated, not only with sea-water, obtained off the Norfolk coast, both sterilized and unsterilized, but also when it was incubated with several of the principal saline constituents of sea-water. The chief result of Purvis and Coleman's varied experiments was to show that the organic substances of sewage when in sea-water only slowly decomposed; for even after eight weeks' incubation there was a comparatively small decrease in the amounts of the two ammonias; and, what is perhaps of greater importance, neither nitrates nor nitrites were produced. In fact, in two series of experiments where the original sewage contained traces of nitrates and nitrites, even these traces disappeared after three days' incubation.

In a later paper by Purvis and Courtauld[2] two series of

[1] *Jour. Roy. San. Inst.* vol. XXVII, No. 8 (1906), p. 433.
[2] *Proc. Camb. Phil. Soc.* vol. XV, Part IV, p. 354.

experiments were conducted by incubating unfiltered sewage with (*a*) ammonia-free water made faintly alkaline with soda, and (*b*) with distilled water, and of such strength that each solution contained 1 per cent. sewage. The general results of these experiments were comparable in some respects with those of Purvis and Coleman in so far as there was a very slow and gradual decrease in the two ammonias. But with regard to the production of nitrates there was some difference, in that both in the alkaline and non-alkaline mixtures very small quantities of nitrates were produced, and in one or two experiments there were also traces of nitrites. In these experiments the zinc-copper couple method as described by Dr McGowan was used to determine nitrates and nitrites. At the same time, attention was called to the possibility that this method might give fictitious results. It was used by Dr McGowan (*Report of Royal Commission on Sewage Disposal*, vol. IV, pt V, p. 17), as well as by Adeney and Letts. It was suggested that such a couple might not only reduce the nitrates and nitrites to free ammonia, but that nitrogenous compounds, like the albuminoids, might be partly reduced to simple forms, of which ammonia would be one; and that, consequently, on distillation the figure obtained for nitrates would be correspondingly high.

In order to substantiate this suggestion, Purvis and Courtauld, some months later (*Proc. Camb. Phil. Soc.* vol. XIV, pt V, p. 441) showed that such a zinc-copper couple did actually reduce peptones, blood serum, albumin and sewage containing no nitrates or nitrites when in solution in strengths varying from 1 per cent. to 0·001 per cent. of the various organic substances. The investigation was divided into three parts: (1) spontaneous decomposition of the dissolved organic nitrogenous substances; (2) the action of a copper-zinc couple upon such solutions; and (3) the action of the couple on these solutions when a definite amount of potassium nitrate was also present. The conclusions were that the couple not only reduced the nitrates to ammonia, but also, partly, the nitrogenous substances as well; and that, therefore, the accuracy of this method could not be relied upon for estimating nitrates and nitrites in water charged with organic nitrogenous substances.

Purvis and Coleman used the indigo method described on p. 10, for nitrates, and the metaphenylene diamine method for nitrites (p. 11). In the later experiments, the phenol sulphuric acid method was used for estimating nitrates (p. 9).

In an investigation by Purvis, Macalister, and Minnett (*Proc. 7th Internat. Congress Applied Chemistry*, 1909, Sec. VIII a, p. 272), the subject was further studied, bacterially as well as chemically. The series of experiments was undertaken to see if stronger sewage and stronger mixtures would indicate the presence of nitrates and nitrites better than in the weaker sewage and weaker mixtures. The following Table (I) contains the results, in parts per 100,000, of the analysis of a 10 per cent. mixture of sewage with sea-water :

TABLE I.

10 per cent. strong afternoon sewage in sea-water ; mixed 2.3.09.

Date of analysis	Free NH_3	Albuminoid NH_3.	Total NH_3	Nitrites	Nitrates
Filtered—					
2.3.09 ..	0·760	0·144	0·904	not done	not done
10.3.09 ..	0·450	0·024	0·474	,,	,,
20.3.09 ..	0·600	0·048	0·648	,,	,,
Unfiltered—					
2.3.09 ..	1·200	0·200	1·400	0·021	none
3.3.09 ..	not done	not done	—	0·018	,,
4.3.09 ..	,,	,,	—	0·011	,,
5.3.09 ..	,,	,,	—	0·009	,,
6.3.09 ..	,,	,,	—	none	,,
10.3.09 ..	0·500	0·300	0·800	,,	,,
20.3.09 ..	0·640	—	—	not done	not done

The following Table (II) contains the estimations of the nitrites and nitrates in the 5 per cent. and 1 per cent. mixtures of unfiltered sewage and sea-water mixed 2.3.09 :

TABLE II.

Date			Nitrites	Nitrates
3.3.09 ..	5 per cent. mixture		0·014	none
4.3.09 ..	,,	,,	0·005	,,
5.3.09 ..	,,	,,	0·004	,,
6.3.09 ..	,,	,,	none	,,
10.3.09 ..	,,	,,	,,	,,
3.3.09 ..	1 per cent. mixture		0·002	,,
4.3.09 ..	,,	,,	none	,,
5.3.09 ..	,,	,,	,,	,,
6.3.09 ..	,,	,,	,,	,,
10.3.09 ..	,,	,,	,,	,,

The chemical results showed: (1) that in all the mixtures of sea-water, with either strong or weak sewage (with variations from 1 to 10 per cent. of sewage), there remained about 70 per cent. of undecomposed and unacted upon nitrogenous organic compounds after 18 days' incubation; (2) that mixtures of 1 per cent. weak sewage and sea-water showed neither nitrates nor nitrites after 18 days; (3) that mixtures of 5 per cent. weak sewage and sea-water showed neither nitrates nor nitrites after 18 days; (4) that mixtures of 10 per cent. *strong* sewage and sea-water gave nitrites for four days and then they disappeared, but that nitrates did not appear; (5) that mixtures of 5 per cent. *strong* sewage and sea-water showed nitrites for three days and then they disappeared, but that no nitrates were produced; and (6) that mixtures of 1 per cent. *strong* sewage and sea-water showed nitrites at the end of the first day of incubation and then they disappeared, but that no nitrates were produced.

The bacterial results of a 10 per cent. mixture of sewage and sea-water proved that there was a rapid diminution of the organisms when the sewage was incubated with sea-water (Tables III, IV, V, pp. 66—67). The rapid disappearance of the bacteria is very marked, and the growths of organisms, for example nitrifying organisms, may be inhibited at a very early stage. In fact the salts in the sea-water act not unlike antiseptics, and the result is that the organic substances are only slowly broken down; and even this decomposition is probably caused by the oxygen dissolved from the air.

Table III.

	Intervals between receipt of sample and bacterial analysis					
	2 hours only		4 days		7 days	
	Sewage	Sewage and sea-water	Sewage	Sewage and sea-water	Sewage	Sewage and sea-water
Colonies in 1 c.c. on agar in 48 hours at 37° C.	26,000,000	2,688,000	39,840,000	3,144,000	8,800,000	36,000
Colonies in 1 c.c. on gelatine in four days at 22° C.	1,072,000,000	11,660,000	250,000,000	206,000,000	188,000,000	1,080,000
B. coli communis in 1 c.c.	1,000,000,000	100,000,000	100,000,000	10,000,000	1,000,000	100
B. ent. spor. in 1 c.c.	1,000	100	1,000	100	100	absent
Strept. fæcalis in 1 c.c.	10,000	1,000	10,000	100	100	absent

Table IV.

The sample was received 3rd February, 1909: a portion was at once diluted in the proportion of sewage 1 part and sea-water 9 parts, *i.e.* a 10 per cent. mixture. Both the original sewage and the mixture were allowed to stand for 12 hours at the laboratory temperature before analysis. The original sewage was diluted with sterile water immediately before analysis to make the results comparable.

	Intervals between receipt of sample and bacterial analysis					
	12 hours after receipt		4 days		7 days	
	Sewage	Sewage and sea-water	Sewage	Sewage and sea-water	Sewage	Sewage and sea-water
No. of colonies in 1 c.c. capable of developing on agar at 37° C. in 48 hours	98,400,000	1,000,000	Not examined	11,600	Not examined	100
No. of colonies in 1 c.c. capable of developing on gelatine at 22° C. in four days ..	Not counted	Not counted		40,160,000		1,200
No. of *B. coli communis* present in 1 c.c.	100,000,000	100,000		10,000		100
No. of *B. ent. spor.* present in 1 c.c. ..	100	nil		nil		nil
No. of *streptococcus fæcalis* in 1 c.c. ..	100	10		nil		nil

The enormous reduction in the number of organisms in seven days is very striking.

Table V.

Sewage received 18th February, 1909, and mixed with sea-water in the proportion of 1 to 9, on receipt; they were allowed to stand at the laboratory temperature for 24 hours and used for analysis on 19th February, 1909.

	Intervals between receipt of sample and bacterial analysis					
	24 hours		4 days		7 days	
	Sewage	Sewage and sea-water	Sewage	Sewage and sea-water	Sewage	Sewage and sea-water
Colonies per c.c. on agar in 48 hours at 37° C.	1,480,000	126,800	268,000	15,600	224,000	2,500
Colonies per c.c. on gelatine in four days at 22° C.	69,200,000	6,200,000	1,024,000	75,600	572,000	32,000
B. coli communis in 1 c.c.	1,000,000	100,000	100,000	100	100,000	100
B. ent. spor. in 1 c.c.	absent	absent	absent	absent	absent	absent
Streptococcus fæcalis in 1 c.c.	100	10	100	10	absent	absent

The decrease in the number of organisms at the end of seven days is again very striking; and the decrease in the number of organisms in the mixture of sea-water and sewage is much greater than the decrease in the sewage. The presence of considerable quantities of *B. coli* is noticeable in the three series of experiments even after seven days.

Purvis, McHattie and Fisher (*Jour. Roy. San. Inst.* 1911, vol. XXXII, p. 442), continued these experiments and during the incubation, dry air, free from carbon dioxide, was slowly and continuously bubbled through mixtures of sea-water and sewage (Tables A and B) and through a mixture of sea-water and distilled water (Table B_2). The numbers are in parts per 100,000.

TABLE A.

10 *per cent. mixture Sewage* (4 *p.m., March 27th,* 1911) *in sea-water. Mixed March 28th,* 1911.

	Mar. 28th	Mar. 29th (1 day)	April 18th (21 days)	May 6th (39 days)	May 19th (52 days)	June 6th (70 days)
Free NH_3	·193	—	·196	·2	·18	traces
Albuminoid NH_3	·052	—	·032	·025	·018	·04
Total NH_3	·245	—	·228	·225	·198	—
N as nitrates	nil	nil	nil	nil	nil	nil
N as nitrites	nil	nil	nil	nil	nil	nil

It was noticed after a few days that there was a dark brown precipitate in the mixture. At the end of 70 days this was filtered, washed, dried, and the nitrogen in it was estimated by Kjeldahl's method. The total nitrogen given was 0·77 part for 100,000 of the original sewage. It appeared therefore, that a considerable amount of nitrogenous material was thrown out of solution. Also it is clear that the diminution in the total ammonia was very small at the end of 52 days, and that no nitrates or nitrites were produced. The method of estimating the nitrates and nitrites in all the incubations was the phenol sulphuric method for the nitrates and the meta-phenylene-diamine method for the nitrites.

TABLE B.

10 *per cent. mixture Sewage* (6 *p.m., April 24th,* 1911) *in sea-water. Mixed April 25th,* 1911.

	April 25th	April 27th (2 days)	April 29th (4 days)	May 6th (11 days)	May 19th (24 days)	June 6th (42 days)
Free NH_3	·252	·214	—	—	·26	·6
Albuminoid NH_3	·03	·036	—	—	·01	·04
Total NH_3	·282	·25	—	—	·27	·64
N as nitrates	nil	nil	nil	nil	nil	nil
N as nitrites	nil	nil	nil	—	—	nil

TABLE B_2.

10 *per cent. mixture Sewage* (6 *p.m., April 24th,* 1911) *in distilled water. Mixed April 25th,* 1911.

	April 25th	April 27th (2 days)	April 29th (4 days)	May 6th (11 days)	May 19th (24 days)	June 6th (42 days)
Free NH_3	·314	·27	—	—	—	·06
Albuminoid NH_3	·03	·036	—	—	—	·06
Total NH_3	·344	·306	—	—	—	·12
N as nitrates	nil	nil	nil	nil	nil	·2
N as nitrites	nil	nil	nil	—	—	nil

It is clear from Tables A and B that, again, there were neither nitrates nor nitrites, whereas in the sewage incubated with distilled water (Table B_2) nitrates appeared at the end of 42 days, but no nitrites were observed. There was an increase in the total ammonia in the sewage and sea-water, but a decrease in the sewage and distilled water incubation. It was also noticed that in both incubations a dark brown precipitate was produced in the sewage and sea-water, and a dark grey in colour flocculent precipitate in the sewage and distilled water. These precipitates were filtered off, washed, and the total nitrogen determined by Kjeldahl's method. The nitrogen obtained from the precipitate of the sewage and sea-water was 1·95 parts per 100,000, and from the precipitates of the sewage and distilled water was 0·98 part. It was therefore obvious that the amount of nitrogenous material thrown down in the sea-water incubation was twice as much as that from the distilled water incubations, although the same sewage and the same strength was investigated during the same time. And what is of importance is to notice the presence of nitric nitrogen in the sewage and distilled water, and its absence in the sewage and sea-water at the end of 42 days.

TABLE C.

5 *per cent. of Sewage* (4 *p.m., April 28th*) *in sea-water. Mixed April 29th,* 1911.

	April 29th	May 6th (7 days)	May 19th (20 days)	June 6th (38 days)
Free NH_3	·207	—	—	·46
Albuminoid NH_3	·015	—	—	·04
Total NH_3	·222	—	—	·5
N as nitrates	nil	nil	nil	nil
N as nitrites	nil	—	—	nil

TABLE D.

5 per cent. of Sewage (4 p.m., April 28th) in NH_3-free water. Mixed April 29th, 1911.

	April 29th	May 6th (7 days)	May 19th (20 days)	June 6th (38 days)
Free NH_3	·083	—	—	traces
Albuminoid NH_3	·001	—	—	·03
Total NH_3	·084	—	—	—
N as nitrates	nil	nil	nil	traces
N as nitrites	nil	—	—	nil

The numbers (Table C, p. 69) again show the increase in the total ammonia, and the absence of nitrates and nitrites at the end of 38 days in the sewage and sea-water ; and the trace of nitric nitrogen in the mixture of sewage and ammonia-free water (Table D) at the end of 38 days, and the decrease in the total ammonia.

The results of the preceding observations were therefore : (1) that even after 70 days' incubation of 10 per cent. sewage in sea-water, with every facility for complete æration, there was no production of nitrates or nitrites ; (2) that there were some nitrates produced when the sewage was incubated with distilled water in 42 days ; (3) that the free-ammonia figure was increased in the sewage and sea-water at the end of 42 days, but it decreased in the sewage and distilled water at the end of the same period of time.

The authors suggested that the most obvious explanation of these facts was to assume that the sea-water destroyed the useful nitrifying organisms, as indicated by the bacterial and chemical investigations of Purvis, Macalister, and Minnett (*loc. cit.*). At the same time, some explanation was necessary with regard to the increase in the free ammonia of the sewage and sea-water, as well as the decrease in the ammonia of the sewage and distilled water and the presence of nitrates in the latter. It was suggested that the free and continuous æration oxidized the nitrogenous compounds of the sewage, and slowly decomposed them to less complex compounds, one of which was ammonia, and that this oxidation was continued a stage further in the sewage and distilled water, so that the presence of the nitrates was accompanied by a corresponding decrease in the free ammonia.

With regard to the continuous presence of free ammonia even after 52 days' incubation, it was remarked that it supported the suggestion of Kenwood and Kay-Menzies (*Internat. Congress Applied Chemistry*, 1909, Sec. VIII a, p. 259) as affording a valuable clue to the contamination of sea-water by sewage.

Letts and Richards (*7th Report Royal Commission on Sewage Disposal*, vol. XI, appendices, part 1, pp. 104–113) also showed that nitrates were only produced in mixtures of sewage and sea-water after about 70 days' incubation.

Purvis and Walker completed another series of experiments both chemically and bacterially and mixed the afternoon unfiltered sewage from the Cambridge Sewage Farm with sea-water obtained from off the Norfolk coast near Lowestoft, as in the previous investigations (*Jour. Roy. San. Inst.* 1912, vol. XXXIII (9), p. 368; 1913, vol. XXXIV (1), p. 71).

The mixtures, and the sea-water itself, were placed in three large unstoppered Winchester bottles. The air, freed from carbon dioxide, and washed by passing through ammonia-free water, was aspirated through each bottle by connection with a water pump, and the aspiration of the air went on day and night. The average temperature was between 14°–15° C. The following tables contain the results of the analyses under the conditions of this constant temperature and continuous and thorough æration, and they give the number of days after the mixtures had been made. The numbers represent parts per 100,000.

TABLE E.—*Analyses of the sea-water* alone.

	1st May (9 days)	7th May (16 days)	20th May (29 days)	3rd June (42 days)	10th June (49 days)
Free and saline NH_3	not done	not done	not done	not done	0·025
Albuminoid NH_3 ..	,,	,,	,,	,,	0·005
Total NH_3 ..	,,	,,	,,	,,	0·030
N as nitrites ..	nil	nil	nil	·020	trace
N as nitrates ..	nil	nil	nil	nil	nil

TABLE F.—5 *per cent. mixture of sewage in sea-water. Mixed 22nd April*, 1912.

	1st May (9 days)	7th May (16 days)	20th May (29 days)	28th May (37 days)	3rd June (42 days)	10th June (49 days)
Free and saline NH_3 ..	·68	·15	·30	·05	·15	·325
Albuminoid NH_3 ..	·11	·08	·08	·05	·085	·045
Total NH_3	·79	·23	·38	·10	·235	·370
N as nitrites	·0037	nil	·006	? trace	nil	nil
N as nitrates	nil	nil	nil	nil	nil	·02

TABLE G.—10 *per cent. mixture of sewage in sea-water. Mixed 22nd April,* 1912. *There were no nitrates or nitrites in the sewage.*

	1st May (9 days)	7th May (16 days)	20th May (29 days)	28th May (37 days)	3rd June (42 days)	10th June (49 days)
Free and saline NH_3	1·02	1·40	·52	·18	·28	·425
Albuminoid NH_3	·15	·17	·17	·15	·15	·145
Total NH_3 ..	1·17	1·57	·69	·33	·43	·570
N as nitrites ..	·0044	trace	·0118	·0059	·0059	nil
N as nitrates ..	nil	nil	nil	? trace	nil	·05

The chief results of the above analyses are (1) the very slow decomposition and decrease in the two ammonias, even after 49 days' incubation. (2) The appearance of nitrites in the sea-water alone in about 42 days, and their appearance in nine days in the two mixtures; their disappearance, and their reappearance, in about 29 days and their decrease in the 10 per cent. mixtures in 42 days; and their disappearance in the 5 per cent. mixture in the same time. (3) The appearance of nitrates after 49 days in the two mixtures.

It is to be noted also that the changes in each mixture are proportionately the same. For example, the total ammonia in both mixtures at the end of 49 days is about one-half that at the end of nine days; and the nitrites in the 5 per cent. mixture are about one-half those in the 10 per cent. mixture at the end of 29 days; and the nitrates in the 5 per cent. mixture at the end of 49 days are about one-half those in the 10 per cent. mixture. The numbers indicate that the conditions of the experiments were very similar.

Letts and Richards (*loc. cit.*) found oxidized nitrogen after 21 days and 27 days in a 5 per cent. mixture, but they did not differentiate it into nitrites and nitrates. They also found both nitrites and nitrates after 73 and 103 days: "the nitrites gradually disappearing and the nitrates becoming stronger, so that after five and half months every trace of nitrous nitrogen had disappeared while nitric nitrogen had largely replaced it."

The bacterial investigations were limited to the number of organisms which appeared on agar at 37° C., and on gelatine at 22° C. The following (Table D_2) contains the bacterial results of the sea-water and the mixtures, of which the chemical

results are contained in Tables E, F and G (pp. 71, 72). The dates represent the days of the month when the incubations on agar and gelatine were commenced.

TABLE D_2.

The sea-water and the two mixtures were analysed at once on the 22nd April, 1912, and then at the intervals which are noted. The numbers represent bacteria per c.c. E = sea-water alone. F = 5 per cent. sewage and sea-water. G = 10 per cent. sewage and sea-water.

		Number of organisms in 1 c.c. capable of developing on agar at 37° C. in 2 days	Number of organisms in 1 c.c. capable of developing on gelatine at 22° C. in 3 days
22nd April, 1912 ..	E	6,000	400,000
	F	1,200,000	4,820,000
	G	2,500,000	10,200,000
26th April, 1912 ..	E	6,400	550,000
(4 days)	F	1,840,000	11,200,000
	G	3,350,000	27,650,000
30th April, 1912 ..	E	740	86,000
(8 days)	F	17,800	1,050,000
	G	29,750	2,850,000
5th May, 1912 ..	E	254	2,550
(13 days)	F	11,400	72,000
	G	20,800	114,000
13th May, 1912 ..	E	84	480
(21 days)	F	942	674
	G	1,380	820
20th May, 1912 ..	E	3	82
(29 days)	F	109	82
	G	220	90
28th May, 1912 ..	E	2	42
(37 days)	F	74	55
	G	93	63
3rd June, 1912 ..	E	53	14
(42 days)	F	87	102
	G	83	276
10th June, 1912 ..	E	71	31
(49 days)	F	94	346
	G	78	1,000

The results of the bacterial investigation are: (1) In the first place they show that a certain number of organisms were in the sea-water itself, although, of course, very considerably fewer than in the mixtures. (2) There was an enormous reduction of the organisms by the 21st day. (3) This reduction steadily went on until the 37th day, and the number growing on agar at 37° was greater than on gelatine at 22° C., whereas

it was the reverse of this at the commencement. (4) That by the 42nd and 49th day there was a gradual increase in the number of organisms.

The experiments were continued after the 49th day, and the effect of the oxidation of *sterilized* sewage and of sterilized sewage mixed with small quantities of the incubations just described was also investigated. The continuous æration at a definite temperature was the same as described before.

The following tables contain the results of the chemical analyses under the conditions of a temperature between 14°–18° C., and of continuous and constant æration.

TABLE E[1].—*Sea-water alone. The numbers represent parts per* 100,000.

	22nd June (61 days)	6th July (75 days)	20th July (89 days)	6th Aug. (106 days)	15th Aug. (115 days)	4th Oct. (165 days)	15th Oct. (176 days)
Free and saline NH_3	·075	·08	·017	·03	·016	·02	·015
Albuminoid NH_3 ..	·0065	·04	·010	·022	·020	trace	·070
Total NH_3	·0815	·12	·027	·052	·036	·02	·085
N as nitrites ..	·0236	nil	·021	·020	nil	nil	nil
N as nitrates ..	? trace	nil	nil	nil	? ·0025	nil	trace

TABLE F[1].—5 *per cent. mixture of sewage in sea-water.*

	22nd June (61 days)	6th July (75 days)	20th July (89 days)	6th Aug. (106 days)	15th Aug. (115 days)	4th Oct. (165 days)	15th Oct. (176 days)
Free and saline NH_3	·03	·15	·050	·150	·050	·15	·13
Albuminoid NH_3 ..	·10	·10	·100	·075	·078	·002	·13
Total NH_3	·13	·25	·150	·225	·128	·152	·26
N as nitrites ..	·0236	nil	·018	·018	very small trace	nil	nil
N as nitrates ..	? trace	·25	trace	trace	·004	?trace	·075

TABLE G[1].—10 *per cent. mixture of sewage in sea-water.*

	22nd June (61 days)	6th July (75 days)	20th July (89 days)	6th Aug. (106 days)	15th Aug. (115 days)	4th. Oct. (165 days)	15th Oct. (176 days)
Free and saline NH_3	·030	·20	·150	·170	·130	·05	·20
Albuminoid NH_3 ..	·180	·15	·130	·160	·330	—	·08
Total NH_3	·210	·35	·280	·330	·460	—	·28
N as nitrites ..	·0295	trace	·015	·020	trace	nil	nil
N as nitrates ..	·0370	1·25	·050	·05	·005	·15	·25

The outstanding results of the above analyses show that (1) the trace of nitrites found in the sea-water alone on the 42nd day remained practically the same on the 61st, the 89th, and the 106th day; and that by the 115th day a trace of nitrates appeared, and also on the 176th day: (2) that there

was an alternate slow disappearance and reappearance of both nitrites and nitrates in the two mixtures between the 42nd day and the 115th day; and that by the 176th day both mixtures contained no nitrites, but definite amounts of nitrates: (3) that the sea-water and mixtures contained definite amounts of both ammonias at the end of the 176th day.

The following table contains the results of the bacterial analyses, which, as in the earlier experiments, were limited to the number of organisms which appeared on agar at 37° C. and on gelatine at 22° C.

E^1 = sea-water alone.
F^1 = 5 per cent. mixture of sewage and sea-water.
G^1 = 10 per cent. mixture of sewage and sea-water.

		Number of organisms in 1 c.c. capable of developing on agar at 37° C. in 2 days	Number of organisms in 1 c.c. capable of developing on gelatine at 22° C. in 3 days
22 June, 1912 (61 days)	E^1	62	17
	F^1	87	287
	G^1	65	760
9 July, 1912 (78 days)	E^1	43	22
	F^1	75	159
	G^1	67	423
22 July, 1912 (89 days)	E^1	31	7
	F^1	39	129
	G^1	58	317
6 August, 1912 (106 days)	E^1	19	2
	F^1	33	81
	G^1	47	208
15 October, 1912 (176 days)	E^1	A few moulds only	A few moulds only
	F^1	A few moulds	A few moulds
	G^1	A few moulds	A few moulds and 7 colonies

The outstanding results of the bacterial analyses were the steady decrease in the number of organisms from the slight increase on the 42nd and on the 49th days; and the final disappearance of all the bacteria by the 176th day, with the exception of seven colonies developed in gelatine in the 10 per cent. mixture. There was nothing but moulds left. A comparison of the corresponding chemical results shows that nitrates were present in the mixture by this time.

Sterilized sewage. Another part of the investigation was to incubate definite portions of sterilized sewage and of sterilized sewage mixed with definite amounts of the 5 per cent. and

10 per cent. mixtures, in order to ascertain how far the decomposition and nitrification are affected in the richer sterilized sewage.

About one litre of fresh sewage was sterilized in an autoclave at 120° C. and 126 lbs. pressure for one hour. Definite volumes of this sterilized sewage were then mixed in the following proportions:

1. 500 c.c. of sterilized sewage were mixed with 500 c.c. sterilized distilled water. This is referred to in H in the tables below.

2. 50 c.c. of the sterilized sewage were mixed with 50 c.c. of the 5 per cent. mixture of sewage and sea-water of the earlier incubations, and also with 900 c.c. of sterilized distilled water. This is referred to in J in the tables below.

3. 100 c.c. of sterilized sewage were mixed with 100 c.c. of the 10 per cent. mixture of sewage and sea-water of the earlier incubations, and also with 800 c.c. sterilized distilled water. This is referred to in K in the tables below.

These mixtures were made on the 27th September, 1912, and allowed to incubate as in the earlier incubations. That is to say, pure filtered air was aspirated regularly and continuously through them at a definite temperature of 14°–18° C., and they were analysed from time to time chemically and bacterially.

TABLE H.—*Chemical Analysis of the sterilized sewage (see above) in parts per* 100,000.

	28th Sept.	4th Oct. (5 days)	10th Oct. (11 days)	15th Oct. (16 days)	20th Oct. (21 days)	5th Nov. (36 days)
Free and saline NH_3	1·95	1·95	1·80	not done	·30	·30
Albuminoid NH_3 ..	·20	·20	·25	,,	·20	·25
Total NH_3	2·15	2·15	2·05	,,	·50	·55
N as nitrites ..	nil	·059	·09	·147	·251	·225
N as nitrates ..	nil	nil	? trace	·150	·450	·900

TABLE J. *Chemical Analysis of the sterilized sewage to which a portion of the old 5 per cent. mixture had been added (see above).*

	28th Sept.	4th Oct. (5 days)	10th Oct. (11 days)	15th Oct. (16 days)	20th Oct. (21 days)	4th Nov. (36 days)
Free and saline NH_3	3·0	1·8	1·4	not done	·15	·08
Albuminoid NH_3 ..	1·0	1·0	1·0	,,	·08	nil
Total NH_3	4·0	2·8	2·4	,,	·23	·98
N as nitrites ..	small trace	·004	·06	·009	? trace	·0001
N as nitrates ..	? trace	nil	? trace	·002	·05	·09

TABLE K.—*Chemical Analysis of the sterilized sewage to which a portion of the old* 10 *per cent. mixture had been added* (*see p.* 76).

	28th Sept.	4th Oct. (5 days)	10th Oct. (11 days)	15th Oct. (16 days)	20th Oct. (21 days)	4th Nov. (36 days)
Free and saline NH_3	5·5	6·0	5·4	not done	·2	·13
Albuminoid NH_3 ..	1·0	1·5	2·2	,,	·15	·05
Total NH_3	6·5	7·5	7·7	,,	·35	·18
N as nitrites ..	trace	·0118	·13	·005	? trace	·0003
N as nitrates ..	? trace	? trace	·10	·200	·45	·75

The results contained in the above tables H, J and K clearly show that nitrites were first produced slowly in all three solutions, and that this was followed by the production of nitrates. At the same time the total ammonia figure is also reduced. That is to say, the oxidation of sterilized sewage resulted in the production of nitrites and nitrates (without the assistance of bacteria), as shown by Table H.

The following table contains the results of the bacterial analyses of which the chemical analyses are in Tables H, J, K (pp. 76–77).

		No. of organisms in 1 c.c. capable of developing on agar at 37° C. in 2 days	No. of organisms in 1 c.c. capable of developing on gelatine at 22° C. in 3 days
29 Sept. 1912	H	No bacterial colonies ; 2 colonies of proteus	No bacterial colonies ; 1 mould
	J	1 colony	No colonies ; 3 moulds
	K	7 colonies	11 ,, no moulds
7 Oct. 1912	H	1 colony	No colonies ; 1 proteus
	J	11 colonies	224 ,,
	K	35 ,,	368 ,,
10 Oct. 1912	H	No colonies	1 colony
	J	11 colonies	218 colonies
	K	19 ,,	320 ,,
15 Oct. 1912	H	No colonies	No colonies
	J	2 colonies	93 colonies
	K	8 ,,	215 ,,
22 Oct. 1912	H	No colonies	No colonies
	J	1 colony	27 colonies
	K	11 colonies	148 ,,
4 Nov. 1912	H	No colonies	No colonies
	J	1 colony	18 colonies
	K	3 colonies	107 ,,

The most striking result of the above table is to show that no bacterial colonies appeared in the sterilized sewage ; and that in the sterilized sewage which had been inoculated with small quantities of the old 5 per cent. and 10 per cent. incubations the number of colonies slightly increased between the date of the inoculation (28th September) and nine days afterwards, and gradually decreased after that date.

If these numbers are compared with the chemical analyses of the 5th November (Table H, p. 76) it will be seen that there were considerable amounts of nitrites and nitrates in the sterilized sewage, and *that not a single bacterial colony was found.*

Results. To sum up the results of these investigations, it is clear that whilst nitrites and nitrates were produced there was also a reduction in the number of bacteria. The first series of experiments with unsterilized sewage indicated that, although the number of organisms decreased, the oxidation of the nitrogenous compounds was not diminished. In the second series of experiments with sterilized sewage, it was proved that nitrites and nitrates were produced even when no bacteria were present ; and although there was a slight increase in the number of organisms in the 5 per cent. and 10 per cent. incubations, no doubt caused by the fresh food which had been added, the numbers gradually decreased, and yet nitrites and nitrates were present in considerable amounts.

The whole series of experiments proves that where thorough æration goes on in mixtures of sewage and sea-water, the production of nitrites and nitrates need not be brought about by the action of the nitrifying organisms. It is a reasonable explanation to suggest that the dissolved oxygen slowly breaks down the complex compounds in sewage ; and that, in course of time, nitrites and nitrates are amongst the final products of this slow oxidation. The salts in the sea-water are antagonistic to the growth of bacteria, the latter gradually die out, and amongst them, it may be, are the useful nitrifying organisms. The result is that a rapid oxidation of the complex chemical compounds is impossible ; and it is only slowly brought about partly by the dissolved oxygen obtained from the air which is gradually used up in the process, and is replenished by the systematic solution of more oxygen from the atmosphere, and partly obtained from aquatic plants which, under the influence of sunshine, give out considerable quantities of oxygen.

The effect of the high temperature and pressure in the sterilized sewage experiments would be to break down the organic compounds to a considerable extent, and thereby the oxidation would proceed more rapidly than in the unsterilized sewage.

The application of these results to the disposal of sewage in the sea or in estuaries or lakes or rivers is direct. The growth and development of useful nitrifying organisms are retarded; and the decomposition and breakdown of the complex organic compounds are slowly accomplished by the oxygen dissolved in the larger body of water. This oxygen is soon used up by the first lot of sewage; and it is only by the continuous solution of more oxygen that the decomposition can proceed. Consequently, the final production of nitrates in this way is a very slow process. An important point to bear in mind is, therefore, the necessity of a thorough and rapid dilution of the sewage or effluent when these are poured into the waters of the sea or an estuary or a river, even after they have undergone some preliminary treatment. A more rapid and efficient oxidation is thereby assured, quite apart from the further purification which proceeds from the ability of aquatic plants to absorb ammonia salts, as well as nitrates. And, of course, a moving water, like the sea, or a river, will also assist the purification.

REFERENCES TO INVESTIGATIONS ON THE DECOMPOSITION OF SEWAGE IN SEA-WATER.

1. Royal Commission on Sewage Disposal, vol. VII. Evidence of Dr Adeney and Prof. Letts.
2. Royal Commission on Sewage Disposal. *5th Report.* Appendix VI. Report by Prof. Letts and Dr Adeney; and the same report and appendix, experiments by Dr Fowler.
3. Purvis and Coleman. *Jour. Roy. San. Inst.* vol. XXVII, No. 8 (1906), p. 433.
4. Purvis and Courtauld. *Proc. Camb. Phil. Soc.* vol. XIV, p. 441, and vol. XV, p. 354.
5. Purvis, Macalister and Minnett. *Proc. 7th Int. Congress.* Applied Chemistry (1909), Sec. VIII *a*, p. 272.
6. Purvis, McHattie and Fisher. *Jour. Roy. San. Inst.* vol. XXXII, No. 9 (1911), p. 442.
7. Royal Commission on Sewage Disposal. *7th Report,* vol. II. Appendices, Part I. Experiments by Prof. Letts and Mr Richards.
8. Letts. *Jour. Roy. San. Inst.* vol. XXXIII, No. 1, p. 11.
9. Purvis and Walker. *Jour. Roy. San. Inst.* vol. XXXIII, No. 9 (1912), p. 368; and vol. XXXIV, No. 1 (1913), p. 71.

The whole subject of "The principles and practice of the dilution method of Sewage Disposal" will be discussed by Dr W. E. Adeney in one of the volumes of The Cambridge Public Health Series.

CHAPTER II

MILK—CREAM—CONDENSED MILK

MILK.

Milk is the fluid secreted by the mammary glands of all female mammals for the purpose of nourishing their young. It is a perfect food and is composed of fat, proteids, mineral salts and sugar. Milk varies slightly in composition, and the milk of the cow, is, of course, of the greatest importance as a food.

Genuine cow's milk has a specific gravity of between 1027·5 and 1035·0, and is slightly yellow in colour, due to the suspended fat. The proteids of milk consist of casein, albumin, globulin, fibrin, and other nitrogenous substances; the mineral matter consisting of the chlorides and phosphates of potassium, sodium, calcium and magnesium, with traces of iron and sulphates. When drawn from the cow it is amphoteric in reaction, *i.e.* it turns red litmus blue, and blue litmus red; but it soon becomes faintly acid owing to the formation of small quantities of lactic acid. This is produced by a micro-organism, *Bacillus acidi lactici*, which gradually decomposes the lactose in milk with the formation of lactic acid. The acid causes the milk to curdle or become "sour," and the length of time required to produce this curdling of milk is chiefly dependent on the temperature. It is well known that milk becomes sour more quickly in summer than in winter.

Milk is undoubtedly the most adulterated of all articles of food. The chief methods of adulteration are:

(*a*) The removal of a portion of the fat or cream,
(*b*) The addition of water,
(*c*) The addition of skimmed or separated milk.

The composition of milk, as sold in this country, is controlled by the Sale of Milk Regulations (1901), issued by the Board of Agriculture, in which it is directed that "Where a sample of milk (not being sold as skimmed or separated or

condensed milk) contains less than 3 per cent. of fat, it shall be presumed...that the milk is not genuine by reason of the abstraction therefrom of milk-fat or the addition thereto of water" and that "Where a sample of milk (not being sold as skimmed, separated or condensed milk) contains less than 8·5 per cent. of milk solids other than milk fat, it shall be presumed...that the milk is not genuine by reason of the abstraction therefrom of milk solids other than milk fat or the addition thereto of water." This standard must be regarded as unduly lenient, as regards the fat content of genuine milk. It is a fact that no healthy, properly-fed cow will produce milk containing as little as 3 per cent. of fat. This is recognized in other countries; as, for example, in the United States of America, where the fat standard is 3·25 per cent.; in the State of Western Australia, where the standard is 3·2 per cent.; in Paris, where the standard is 4·0 per cent., and in Berne, where the standard is 3·5 per cent.

Richmond has given in the *Analyst*, vols. XXVIII to XXXVII, the average fat in the samples of milk examined in the Laboratory of the Aylesbury Dairy Company, and his results in percentages for the last ten years are given below.

	1903	1904	1905	1906	1907	1908	1909	1910	1911	1912
Fat	3·83	3·74	3·73	3·71	3·75	3·75	3·74	3·73	3·71	3·68
Solids, not fat ..	8·95	8·94	8·97	8·93	8·94	8·88	8·92	8·89	8·80	8·86

These numbers represent the results of the analyses of many thousands of milks annually.

Determination of the Specific Gravity. The sample is first carefully shaken or stirred thoroughly to mix any cream which may have risen to the top, and great care should be taken to avoid the inclosure of air, as milk retains air bubbles rather tenaciously.

The specific gravity of the milk is taken at 60° F. (16° C.) and the sample should be brought to this temperature by allowing it to stand in water. The specific gravity may be determined either with a delicate hydrometer, a Westphal balance or with a specific gravity bottle. The two former read the specific gravity directly, but the specific gravity bottle method is the most accurate. The specific gravity of normal

milk, which varies from 1027·5 to 1035·0, is lowered by the addition of water and raised by skimming.

Example: the weight of water contained in a specific gravity bottle was 49·987 grams.

The weight of a sample of milk contained in the same bottle was 51·051 grams.

The specific gravity of the sample therefore was

$$\frac{51{\cdot}051}{49{\cdot}987} = 1030{\cdot}56.$$

Estimation of total solid matter. 5 grams of the sample are weighed out into a flat bottomed platinum dish, evaporated on the water bath to dryness, and, when dry, placed in a water oven (at 100° C.). It is allowed to cool in a desiccator and weighed from time to time until the weight is constant. From two to three hours in the water oven may be required.

Example: The residue from 5 grams of milk was 0·613; therefore the total solids in the milk were

$$0{\cdot}613 \times 20 = 12{\cdot}26 \text{ per cent.}$$

Ash. The residue from the determination of the total solids is carefully incinerated over a Bunsen flame until a white ash remains. Great care should be taken that the temperature does not rise too high otherwise the result will be low owing to the volatilization of some of the sodium chloride. The ash of milk averages about 0·75 per cent., and it is slightly alkaline. If the alkalinity is marked it would probably indicate the addition of carbonate in the form, perhaps, of calcium carbonate or chalk.

Fat. There are many methods for the determination of fat in milk; but for all practical purposes the following will suffice:

The Leffman-Beam Process. This well-known method is the most accurate and the most convenient of all centrifugal methods. The results obtained with it, by a skilled worker, never vary more than 0·1 per cent. from the most accurate methods.

The process depends upon the liberation of the fat by the

addition of concentrated sulphuric acid, and its separation by the application of centrifugal force. Before the liberation of the fat a mixture of fusel oil and hydrochloric acid is added which produces a greater difference in the surface tension between the fat and acid liquid and this causes a readier separation of the fat.

The bottles used in the process are flat bottomed and hold about 30 c.c. ; they are graduated on the neck into 80 divisions, each division representing 0·1 per cent. of fat by weight.

15 c.c. of the milk are measured into the bottle, 3 c.c. of a mixture of equal parts of amyl alcohol and concentrated hydrochloric acid are added and the liquids well mixed ; 9 c.c. of concentrated sulphuric acid are then added slowly with constant agitation, and then the bottle is filled almost to the zero mark with a hot mixture of equal parts of sulphuric acid and water. It is then placed in the centrifugal machine (if one test only is being made, the machine must be balanced with a bottle filled with equal parts of sulphuric acid and water) and rotated for two minutes. After the rotation, the fat will have risen to the top of the bottle, and the percentage is read off directly. The reading must be made from the extreme top to the extreme bottom of the column of fat.

The Werner-Schmidt Process. This method is particularly suitable for sour milks. 10 grams of the sample are placed in a special tube (which is graduated from 20 to 50 c.c.), and the tube is filled to the 20 c.c. mark with concentrated hydrochloric acid. The contents of the tube are well mixed and the tube is put in a beaker of water which is heated to boiling until the contents turn dark brown. The tube is allowed to stand for about five minutes and afterwards cooled in water ; pure ether is then added to the 50 c.c. mark, the tube corked and shaken for one minute. After shaking, let the tube stand until the acid and ether layers have completely separated. The volume of the ether layer is read off and 20 c.c. are transferred into a weighed fat flask, by means of a pipette. The ether is evaporated and the fat dried in the water oven for 45 minutes and weighed.

Example : 10 grams of milk were taken for the estimation ;

the volume of the ethereal layer was 25·5 c.c., the weight of fat obtained from 20 c.c. was 0·253 gram, then

$$\frac{0{\cdot}253 \times 25{\cdot}5 \times 10}{20} = 3{\cdot}225 \text{ per cent. of fat.}$$

The Adams' process. This is a general method of extraction and a Soxhlet apparatus is used. With regard to the extraction of the fat in milk, 5 c.c. are run over one of the long strips of fat-free paper specially manufactured for the process. The paper is then rolled into a coil and dried in a water oven. When it is completely dried, place it in the Soxhlet apparatus attached to a weighed dry flask. Pure ether is then run in from the top of the inverted condenser in sufficient amount to fill the Soxhlet tube about one and a half times. Place the flask on a water bath and heat with a small flame. The ether should be allowed to siphon over about a dozen times when it will have dissolved the whole of the fat. The ether is distilled over at the end of the siphonings ; the flask detached and dried in the water oven until the weight is constant. This weight, less the weight of the flask, represents the weight of fat in 5 c.c. of the milk, from which the percentage can be calculated.

Example : 5 c.c. of milk gave 0·165 gram of fat : the specific gravity of the milk was 1031, therefore the 5 c.c. weighs 5·155 : hence

$$\frac{100 \times 0{\cdot}165}{5{\cdot}155} = 3{\cdot}2 \text{ per cent. of fat.}$$

The fat-free papers used in this process usually contain small quantities of substances which are soluble in ether. It is desirable, therefore, that the papers should be extracted with ether first, and then dried, otherwise the results will be too high.

The Gottlieb-Rose Method (*Analyst*, 1898, XXIII, 259). Place 5 grams of milk in a 50 c.c. stoppered cylinder ; add 0·5 c.c. of ammonia (specific gravity 0·880), and thoroughly mix the contents of the tube ; then add 5 c.c. of alcohol (97 per cent.) to the mixture and again mix the contents of the tube ; to the mixture add 12·5 c.c. of ether and mix the liquid, followed by 12·5 c.c. of petroleum ether and again thoroughly mix ; allow

the liquids to separate and, by means of a wash-bottle tube, blow off the clear liquid containing the fat into a weighed fat flask; the liquid remaining in the cylinder is again shaken up twice with 25 c.c., and once with 20 c.c. of the mixed solvents, and the washings are each in turn blown over into the same flask. The solvents are evaporated; the flask dried in a water oven and weighed; the weight of fat obtained multiplied by 20 gives the percentage of fat.

Calculation Method. Several formulæ have been proposed by which the third factor may be calculated when any two are known. Richmond's formula has been found to be the most satisfactory and is in almost universal use:

$$T = 0{\cdot}25G + 1{\cdot}2F + 0{\cdot}14,$$

where T = total solids, G = the excess specific gravity over 1000, and F = the percentage of fat.

Upon this formula, Richmond's slide rule has been based, by which the fat may be read off when the specific gravity and total solids are known, or the total solids when the fat and specific gravity are known.

Total Proteids. These are best determined by calculation from the total nitrogen obtained by the Kjeldahl method.

Solutions required: Concentrated sulphuric acid.

Strong sodium hydroxide solution. 50 grams of sodium hydroxide are dissolved in 50 c.c. of water and the solution is allowed to stand until it is clear; the clear liquid is poured off and kept in a rubber stoppered bottle.

N/10 sulphuric acid solution.

N/10 sodium hydroxide solution.

Weigh out 5 grams of milk into a Kjeldahl flask and evaporate to dryness on a water bath; to the dry residue add 5 grams of powdered potassium sulphate, and 20 c.c. of concentrated sulphuric acid; the flask is then placed in an inclined position and heated gently with a naked flame until all the frothing ceases; the flame is then raised until the acid boils briskly. As soon as the liquid is colourless it is allowed to cool, and is then rinsed out into a distilling flask, about 200 c.c. of water being used for this purpose. Strong sodium hydroxide

solution is added in sufficient quantity to make the liquid alkaline and also a few pieces of pumice ; the flask is at once connected to the condenser. Into the receiving flask, place 50 c.c. of the N/10 sulphuric acid solution, which is to absorb the ammonia as it distills over. The distillation is continued until about 200 c.c. have passed over ; the excess of sulphuric acid in the receiver is then titrated with the N/10 sodium hydroxide solution. Each c.c. of N/10 sulphuric acid solution used = 0·0014 gram of nitrogen.

The total proteids are obtained by multiplying the amount of nitrogen by 6·38.

A blank experiment should be conducted with the materials used.

In this method organic nitrogen is converted into ammonia, and the ammonia combines with the sulphuric acid to form ammonium sulphate. This is decomposed by the sodium hydroxide solution, and the liberated ammonia is distilled over and collected in the N/10 sulphuric acid solution in the receiver.

Example : In a determination of the total proteids, 5 grams of milk were used and 21·4 c.c. of the N/10 sulphuric acid solution were neutralized by the ammonia formed.

Then $21{\cdot}4 \times 0{\cdot}0014 \times 20 \times 6{\cdot}38 = 3{\cdot}823$ per cent. of total proteid.

The total proteid may also be determined approximately by calculation from Richmond's formula

$$P = 2{\cdot}8T + 2{\cdot}5A - 3{\cdot}33F - 0{\cdot}7\frac{G}{D}.$$

P = the percentage of proteid.
T = the percentage of total solids.
A = the percentage of ash.
F = the percentage of fat.
D = the specific gravity (water being 1).
G = the excess gravity over 1000.

Milk Sugar (Lactose). Milk sugar may be determined with sufficient accuracy by calculation. The fat, proteids and ash subtracted from the total solids will give the lactose. A slight error is introduced owing to the fact that the ash is not the same as the salts existing in the milk ; and that it is assumed

that everything besides fat, ash and proteid is lactose, a supposition which is not strictly accurate.

A fairly accurate determination of lactose can be determined by Fehling's method.

Solution required: Weigh out 34·6 grams of pure crystallized copper sulphate, which has been crushed and dried between blotting paper; dissolve in distilled water and make up to 500 c.c. Also weigh out 173 grams of rochelle salt and 65 grams of sodium hydroxide; dissolve each separately in distilled water; mix the two solutions and dilute to 500 c.c.

The process. Weigh out 10 grams of milk and dilute to 100 c.c. with distilled water; add a few drops of acetic acid and shake thoroughly well to coagulate the proteids. Neutralize the acetic acid with soda solution, filter from the precipitated proteids; take 10 c.c. of the filtrate and fill up to 100 c.c. with distilled water, so that a 1 per cent. solution of the milk is thus obtained. Place 25 c.c. of the copper solution and 25 c.c. of the rochelle salt and soda solution in a dish; add about 100 to 150 c.c. of water; mix well; raise the temperature so that the solution first simmers (not boils) and run in the 1 per cent. milk solution from a burette, stirring well all the time. The blue colour of the Fehling's solution will gradually disappear; but it is not easy to see the exact point when all the copper is precipitated as cuprous oxide. In order to do this a series of drops of ferrocyanide of potassium, made very slightly acid with a drop of acetic acid, are placed on a porcelain dish. Towards the end of the experiment a drop of the solution in the dish, taken out at the end of a stirring rod, is brought in contact with the drop of ferrocyanide of potassium. If the copper has not been completely precipitated by the lactose, a chocolate coloured precipitate will be noticed. When this disappears, all the copper in the Fehling's solution has been precipitated. It is then easy to calculate the percentage of sugar, for 50 c.c. of Fehling's solution are equivalent to 0·3380 gram lactose or milk sugar.

The percentage of lactose is also accurately determined by means of the polarimeter after removing the fat and proteids.

The specific rotatory power of a substance means the

amount of rotation in angular degrees of the plane of polarization produced when a ray of polarized light passes through a solution of the substance containing 1 gram of the substance in 1 c.c. in a tube 1 decimetre long; and it is usually expressed for the monochromatic light of a sodium flame as:

$$[\alpha]_D = \frac{100\,\alpha}{l \times c}$$

where α = the observed angle of rotation,

l = the length of the observation tube in decimetres,

c = the concentration per 100 c.c.,

and $[a]_D$ = the angular rotation for lactose and is equal to 52·5°.

Solutions required: Acid mercuric nitrate solution. Dissolve a small quantity of metallic mercury in twice its weight of nitric acid (specific gravity 1·42) and when dissolved, add an equal volume of water.

For the estimation, place 60 c.c. of the milk in a 100 c.c. flask; add 1 c.c. of the acid mercuric nitrate solution and fill the flask to the mark with distilled water; shake the solution well and filter through a dry filter paper; place the filtrate in a polarimeter tube and take several readings of the angular rotation.

In calculating the results, correction must be made for the volume of the precipitated fat and proteids; the weight of the fat multiplied by 1·075 gives its volume; and the volume of the proteids is obtained by multiplying the weight by 0·8; the sum of these two is the volume in c.c. of the precipitate.

Example: The specific gravity of a sample of milk was 1031·0; the percentage of fat 3·5; and the percentage of proteids 3·55.

The weight of milk was 60 c.c. × 1·031 = 61·86 grams.

The weight of fat was $\frac{3{\cdot}5 \times 61{\cdot}68}{100} = 2{\cdot}165$ grams.

The volume of fat was then 2·165 × 1·075 = 2·33 c.c.

The weight of proteids in the milk was

$$\frac{3{\cdot}55 \times 61{\cdot}86}{100} = 2{\cdot}30 \text{ grams.}$$

The volume of proteid was $2{\cdot}30 \times 0{\cdot}8 = 1{\cdot}84$ c.c.

The volume of the solution taken for the determination of the lactose was therefore $100 - (2{\cdot}30 + 1{\cdot}84) = 95{\cdot}86$ c.c.

The angle of rotation in a 200 mm. tube was $2{\cdot}5°$; substituting these values in the above equation we get:

$$52{\cdot}5 = \frac{95{\cdot}86 \times 2{\cdot}5}{c \times 2}$$

or

$$c = 2{\cdot}28,$$

that is, there are 2·28 grams of lactose in 61·86 grams of milk or 3·69 per cent. of lactose.

Non-fatty solids. The non-fatty solids are obtained by subtracting the percentage of fat from the percentage of total solids; or by calculation from the fat and specific gravity. The formula for the latter is

$$SNF = \frac{S + F}{4}$$

where SNF = the percentage of non-fatty solids,
S = the specific gravity less 1000,
F = the percentage of fat.

Calculation of adulteration. The calculation of the percentage of added water in a sample of milk is made from the percentage of non-fatty solids. The non-fatty solids in a sample of genuine milk should not fall below 8·5 per cent. For example, V parts of non-fatty solids correspond to $\frac{V \times 100}{8{\cdot}5}$ parts of genuine milk, and the percentage of added water in the sample is found from the formula

$$100 - \frac{V \times 100}{8{\cdot}5}.$$

Example: A sample of milk gave 8·00 per cent. of non-fatty solids then $100 - \frac{8{\cdot}00 \times 100}{8{\cdot}5} = 5{\cdot}88$ per cent. of added water.

Since the minimum percentage of fat in a genuine milk is 3·0, the deficiency in a sample is calculated from the formula

$$100 - \frac{F \times 100}{3}.$$

Example: A sample of milk contains 2·7 per cent. of fat then

$$100 - \frac{2{\cdot}7 \times 100}{3} = 10$$

and the sample is deficient of 10 per cent. of its fat.

Where a sample has been deprived of a portion of its fat and also watered, an allowance must be made in the percentage deficiency in fat for the deficiency caused by the addition of the water.

Example: The results of an analysis of a sample of milk were 2·40 per cent. of fat and 7·65 per cent. of solids not fat; and, calculating on the formula, the sample contains 10 per cent. of added water. The original milk before the water was added contained $\frac{100 \times 2{\cdot}4}{90} = 2{\cdot}66$ per cent. of fat,

and $$100 - \frac{2{\cdot}66 \times 100}{3{\cdot}0} = 11{\cdot}33.$$

The sample, therefore, contained 10 per cent. of added water, and, in addition, was deficient of 11·23 per cent. of its fat.

Analysis of a sour milk. It is often necessary, especially in summer, to undertake the analysis of a sour milk; but, with care, the results should not differ materially from the analysis of the milk before it has gone sour. The greatest difficulty is to obtain an even mixture of the sample; but it is generally possible to obtain this if the whole of the sample is poured into a beaker and mixed with a wire egg beater.

The fat is estimated by the Werner-Schmidt or the Gottlieb-Rose method.

Determination of the total solids. Approximately 5 grams of the well mixed sample are weighed into a platinum dish; one drop of phenol phthalein is added, and then N/10 sodium hydroxide solution drop by drop until a pink colour is just permanent; the sample is then evaporated on a water bath; dried in a water oven and weighed; from the weight is subtracted 0·0022 gram for each c.c. of N/10 sodium hydroxide solution added.

Example: 5·30 grams of a sour milk required 4·5 c.c. of N/10 sodium hydroxide solution to produce a permanent pink

colour ; and, after drying, the residue weighed 0·587 gram ; $4·5 \times 0·0022 = 0·0099$; and therefore,

$$\frac{(0·587 - 0·0099) \times 100}{5·30} = 11·06 \text{ per cent. of total solids.}$$

Estimation of dirt in milk by Lowe's Method (*Chemical News*, 1912, vol. CIV, p. 61). The apparatus required is a conical settling tube to which is attached, by a rubber band, a small tube graduated in 0·1 c.c.; 500 c.c. of the milk are placed in the settling tube and allowed to stand overnight ; a few drops of formalin being added to prevent curdling ; the volume of the sediment is read off in the morning and is reported as parts per 100,000 by volume ; or the sediment may be collected, dried at 100° C. and weighed, in which case the dry residue should be multiplied by eight, as it has been proved that only $\frac{1}{8}$th part by weight of cow dung can be recovered from the milk. The sediment should be carefully examined under the microscope after washing.

Test for cow dung. The sediment is treated with a strong solution of sugar in a watch glass ; the sugar solution is poured off, and the sediment dried at 100° C. ; one drop of concentrated sulphuric acid is now added, and the particles of dung will become a reddish purple colour, which is readily seen with a hand glass.

The sediment should also be examined microscopically for any moulds or bacteria and for any vegetable parenchyma and cells and other dirt.

Skimmed milk.

Skimmed milk is the residue left after the removal of a large proportion of the fat from a milk by hand or by centrifugal means. The removal of the fat causes a slight increase in the proportion of non-fatty solids, and also an increase in the specific gravity.

The sale of skimmed milk in this country is controlled by the Sale of Milk Regulations, 1912, issued by the Board of Agriculture, by which it is directed that " Where a sample of skimmed or separated milk (not being condensed milk) contains less than 8·7 per cent. of milk solids other than milk fat it shall

be presumed...until the contrary is proved, that the milk is not genuine, by reason of either the addition thereto of water or the abstraction therefrom of milk solids other than milk fat."

The analysis of a skimmed milk is carried out exactly as if it were a full cream milk.

Buttermilk.

Buttermilk is the residue left in the churn after the removal of the butter. Its composition varies considerably owing to the fact that a certain amount of water is added to facilitate the separation of the butter, and the amount varies with the temperature. Nevertheless, the addition of 25 per cent. of water should be regarded as exceptional, and anything above 25 per cent. as fraudulent. The analysis of buttermilk is carried out as if it were a sour milk.

Preservatives in Milk.

The presence of any preservative substance in milk is prohibited under the Public Health (Milk and Cream) Regulations, 1912, formulated by the Local Government Board under the provisions of the Public Health (Regulations as to Food) Act, 1907. But preservative substances are to a certain extent still used; those most commonly employed being compounds of boron and formaldehyde.

Detection of boric acid and borax. Place about 5 c.c. of the milk in a porcelain crucible; add six drops of tincture of turmeric and three drops of dilute hydrochloric acid (1 in 3); the mixture is then gently evaporated on the top of a water oven. In the presence of either boric acid or borax, a pink colour is developed at the edges, this colour is characteristic, and the reaction is very delicate.

Estimation of boric acid by Thompson's Method (*Analyst*, 1893, XVIII, p. 184). 50 c.c. of the milk are evaporated to dryness with 1 gram of sodium hydroxide; the residue is incinerated at a low temperature and the ash dissolved in dilute hydrochloric acid; the contents of the dish are washed into a 100 c.c. flask, the volume must not exceed 50 to 55 c.c.; 0·5 gram of dry

calcium chloride is added and one drop of phenol phthalein solution ; sodium hydroxide solution is then run in until the solution is alkaline, and, finally, 20 c.c. of lime water are added and the solution is made up to 100 c.c. In this way, all the phosphates are precipitated as calcium phosphate. The solution is now well mixed and filtered through a dry filter paper ; 50 c.c. of the filtrate (= 25 c.c. of milk) are taken for the estimation, and normal sulphuric acid is added until the pink colour is discharged ; a few drops of methyl orange are then added, and the addition of sulphuric acid is continued until the yellow colour becomes pink ; N/10 sodium hydroxide solution is now added until the pink colour just becomes yellow. All the acids, except boric acid and carbonic acid, now exist as salts neutral to phenol phthalein ; the carbonic acid is expelled by boiling, the solution is cooled, and 30 grams of glycerine are added ; N/10 sodium hydroxide solution is then run in until a permanent pink colour is produced.

1 c.c. N/10 sodium hydroxide = 0·0062 gram of boric acid.

Example : A sample of milk treated in this way required 7·9 c.c. of N/10 sodium hydroxide after the addition of glycerine to produce a permanent pink colour ; therefore

$$7{\cdot}9 \times 0{\cdot}0062 \times 4 \times 700 = 137{\cdot}14$$

grains of boric acid per gallon of milk.

Detection of formaldehyde. One of the best tests is that of Jorissons ; take about 10 c.c. of the milk and add several drops of an aqueous solution of phloro-glucinol ; shake well ; and then add a few drops of sodium hydroxide, a pink colour is produced in the presence of formalin.

Hehner's test. Take 5 c.c. of milk and dilute with about 25 c.c. of water ; add a drop of carbolic acid and then carefully run some strong sulphuric acid down the side of the test tube ; a red ring at the junction of the two solutions indicates the presence of formaldehyde.

On the whole, if formalin is suspected, it is better to distil about two-thirds of 100 c.c. of the milk, and use the distillate for the tests.

Schiff's test. A very dilute solution of magenta is decolourized

with sulphurous acid; add a c.c. of the distillate to this and allow to stand for a few minutes; the colour will be reproduced by traces of formaldehyde.

Estimation of formaldehyde. Solutions required: Acid silver nitrate solution. To 30 c.c. of N/10 silver nitrate add 15 drops of concentrated nitric acid.

N/10 solution of potassium sulphocyanide.

N/1 solution of potassium cyanide containing 6·5 grams per 1000 c.c.

Place 100 c.c. of milk in a distilling flask with 1 c.c. of dilute sulphuric acid (1 in 3), and distil the mixture carefully until 20 c.c. of the distillate have been collected; these 20 c.c. contain approximately one-third of the formaldehyde present in the original 100 c.c. To the distillate add 5 c.c. of the N/1 potassium cyanide solution, and then 10 c.c. of the acid silver nitrate solution; shake the mixture; make it up to 50 c.c.; filter; take out 25 c.c. with a pipette for the estimation; and titrate with the N/10 potassium sulphocyanide solution (Volhard's method).

10 c.c. of the acid silver nitrate solution are then added to 5 c.c. of the N/1 potassium cyanide solution; make up to 50 c.c., well mix and filter; 25 c.c. are taken out and titrated with the N/10 potassium sulphocyanide solution.

The difference in the two titrations is owing to the fact that some of the potassium cyanide forms a double compound with the formaldehyde. Each c.c. of potassium sulphocyanide = 0·006 gram of formaldehyde.

Example: In a milk containing formaldehyde, 2·3 c.c. of potassium sulphocyanide solution were used for 25 c.c. without formaldehyde, and 3·3 c.c. for the 25 c.c. with formaldehyde; the difference being 1·0 c.c. Therefore $1{\cdot}0 \times 2 \times 0{\cdot}006 \times 3 = 0{\cdot}036$ gram of formaldehyde in 100 c.c. of milk.

Jones' method (*Chemical News*, 1908, XCVII, p. 247). *Solutions required: Acid reagent.* Dissolve 0·25 gram of pure iron wire in hydrochloric acid; oxidize the solution with nitric acid and precipitate the iron with ammonia; wash the precipitate and dissolve it in 500 c.c. of concentrated hydrochloric acid.

Dilute formalin solution. 1 c.c. of formalin (40 per cent. formaldehyde) is diluted to 1000 c.c. 0·1 c.c. of this solution added to 10 c.c. of milk gives a product containing one part of formalin per 100,000 parts of milk or one part of formaldehyde per 250,000 parts of milk.

The process. 10 c.c. of the suspected milk in a large test tube (one inch by six inches) are treated with 10 c.c. of the acid reagent. Into each of two similar tubes, 10 c.c. of pure milk are placed; and, to one of these 0·1 c.c. and to the other 0·2 c.c. of the standard formalin solution is added and also 10 c.c. of the acid reagent to each; the contents of the tubes are well mixed; the tubes are placed simultaneously in a water bath at 90° C., and subsequently kept at 80° to 85° C. for 20 to 25 minutes; to each tube 30 c.c. of cold water are added and the mixture well shaken; cooled; filtered; each filter washed with 10 c.c. of water and the filtrates made up to 100 c.c. in each case.

The blue colours which are obtained are compared in Nessler cylinders; if the colour shown in the sample filtrate be darker than that given by the 0·1 c.c. of the standard filtrate, it is better to dilute the 0·2 c.c. standard filtrate. The sample and standards must be submitted to exactly the same conditions side by side.

Detection of mystin. Quite recently a proprietary article called mystin has been used as a preservative with the intention of defeating the usual tests for preservatives. This substance consists of a solution of formaldehyde and sodium nitrite in water; the sodium nitrite is added to prevent the colouration for formaldehyde being obtained in the Hehner test.

Dr Monier Williams (*Local Government Board Food Report*, No. 17, 1912) has shown that if 5 c.c. of a sample of milk containing mystin are treated with 0·05 gram of urea and 1 c.c. of N/1 sulphuric acid in a boiling water bath for two minutes and then cooled, the resulting liquid will give a well defined Hehner colouration.

Detection of benzoates. Sodium benzoate has been used as a preservative of milk. It may be identified by adding a drop or two of acetic acid to the milk; shake well; filter from the

coagulated proteids; neutralize the filtrate carefully with sodium hydroxide and add ferric chloride when a buff coloured precipitate will be produced insoluble in acetic acid if a benzoate is present.

Detection of salicylic acid. A portion of the milk is treated with one or two drops of acetic acid; shake well; add a few drops of mercuric nitrate; filter off the precipitated proteids; shake the filtrate with ether, repeating the extraction several times; and evaporate the extractions to small bulk; add one or two drops of ferric chloride when a violet colour will be produced in the presence of salicylic acid.

(Also see Chap. XI for the detection and estimation of preservatives in foods.)

Typical Analyses of Milk (in percentages).

	(1)	(2)	(3)	(4)	(5)	(6)	(7)
Fat	3·53	2·85	3·20	2·23	7·27	0·60	0·53
Solids, not fat ..	8·91	8·70	8·02	7·52	7·40	7·15	3·68

	(8)	(9)	(10)
Fat	0·20	1·12	5·81
Solids, not fat ..	8·90	8·56	10·25

No. (1) is a genuine milk of good quality.

No. (2) is a milk deficient of 5 per cent. of its fat.

No. (3) is a milk containing 5·5 per cent. of added water.

No. (4) is a milk containing 11·5 per cent. of added water, and deficient in 16 per cent. of its fat.

No. (5) is a sample of milk improperly taken; it was taken from the top of a churn without first mixing the contents.

No. (6) is a genuine sample of buttermilk containing 15·5 per cent. of added water.

No. (7) is a sample of buttermilk containing 56·7 per cent. of added water, an excess of 31·7 per cent.

No. (8) is a genuine sample of skimmed milk.

No. (9) is a typical analysis of fore milk.

No. (10) is a typical analysis of strippings.

The following analyses of milk (in percentages) have been kindly supplied by Dr Laird, the Medical Officer of Health for

Cambridge. The samples were analysed by Mr West Knights, the official Analyst for Cambridge.

	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)
Total solids ..	11·70	10·92	10·68	11·74	11·74	7·82	11·60	9·94
Solids, not fat ..	8·75	7·90	7·98	9·01	8·86	5·47	8·90	6·59
Milk fat.. ..	2·95	3·02	2·70	2·73	2·88	2·35	2·70	3·35
Ash	0·74	0·72	0·60	0·76	0·70	0·48	0·72	0·58

(11) deficient in 2 per cent. of fat; (12) 7 per cent. added water; (13) 6 per cent. added water; (14) deficient in 9 per cent. of fat; (15) deficient in 4 per cent. of fat; (16) adulterated with 35 per cent. water; (17) deficient in 10 per cent. fat; (18) deficient in 22 per cent. non-fatty solids.

The following series of analyses show that the various samples of milk are of a higher quality.

	(19)	(20)	(21)	(22)	(23)	(24)	(25)	(26)
Total solids ..	11·88	11·82	13·24	13·96	11·84	13·60	13·90	12·68
Solids, not fat	8·58	8·17	9·44	9·26	8·54	9·10	9·80	8·18
Milk fat ..	3·30	3·65	3·80	4·70	3·30	4·50	4·10	4·50
Ash	0·72	0·70	0·78	0·74	0·68	0·68	0·80	0·68

The chemical changes produced in boiled and sterilized milk have been investigated by Purvis, Brehaut and McHattie (*Jour. Roy. San. Inst.* 1912, vol. XXXIII, p. 154) who showed that definite amounts of proteids, phosphates and fat are thrown out of solution and form the well-known scum. In round numbers the loss in phosphorus in boiled milk was found to be 11 per cent. and in sterilized milk 4 per cent.; the loss in proteids in the boiled milk was about 14 per cent. and in the sterilized milk about 6 per cent.; and the loss in fat in the boiled milk was about 6 per cent. Besides these losses, it means that definite chemical changes take place in milk when it is either boiled or sterilized.

A full discussion of the natural variations in the composition of cows' milk is contained in the *Departmental Committee's Report on Milk and Cream Regulations*, 1901 (Cd. 491); and a considerable number of analyses are quoted.

The final *Report of the Irish Milk Commission*, 1911 (Cd. 7129) is also of great importance as regards the milk supply and its purity.

CREAM.

Cream is obtained either by allowing milk to stand, when it rises to the top and is removed by hand ; or by the separation of the cream from the milk in a centrifugal separator.

The chief adulterations of cream are deficiency in fat and the addition of thickening substances and preservatives.

The addition of preservatives to cream is now controlled by the Public Health (Milk and Cream) Regulations, 1912, issued under the Public Health Act, 1907. Under these regulations, the addition of thickening substances to any cream, and the addition of preservative substances to cream containing less than 35 per cent. of fat ; or the addition to cream containing more than 35 per cent. of fat of any preservative substance other than boric acid or hydrogen peroxide is prohibited. Further, any cream containing preservative must be described as " Preserved Cream " and the amount of the preservative present must be stated on a label of specified form and size.

Estimation of fat. This can be done by any of the methods used for milk ; but in the Werner-Schmidt process a definite weight of the cream should be diluted with a definite volume of distilled water. In Adams's process the cream can be easily plastered over the long strip of fat-free paper and then dried in the hot water oven.

A fairly accurate determination of the fat in cream may be made by means of the modified Leffman-Beam process for milk. Two grams of the sample are weighed in a flat porcelain dish, and about 5 c.c. of water are added ; the mixture is warmed gently and thoroughly stirred, and poured into one of the Leffman-Beam bottles ; the dish is washed twice with about 4 c.c. of water ; the washings are added to the rest of the liquid in the bottle ; amyl alcohol and hydrochloric acid are then added followed by sulphuric acid as described under milk (pp. 82, 83). The Leffman-Beam bottles are graduated for 15·55 grams of milk so that the percentage of fat read off is multiplied by 7·775.

For a more accurate determination, the Werner-Schmidt method is used; 2 grams of cream are taken for the estimation and diluted with about 8 c.c. of water.

The total solids, ash, sugar and proteids are determined by the methods described under milk (pp. 82, 85, 86).

Test for gelatine. *Solution required:* Acid mercuric nitrate solution which is prepared by dissolving one part of mercury in two parts of concentrated nitric acid; when all the mercury is dissolved the liquid is cooled and diluted with an equal volume of water. 10 c.c. of the milk are placed in a test-tube, diluted with 20 c.c. of water and thoroughly shaken; 10 c.c. of the acid mercuric nitrate solution are added and the mixture is thoroughly shaken for several minutes; the tube is set aside for the proteids to settle; the contents are filtered; an equal volume of saturated picric acid solution is added to the filtrate when, in the presence of gelatine, a flocculent heavy precipitate is formed.

Detection of gelatine in sour cream (A. Seidenberg, *Jour. Ind. Eng. Chem.* 1913, 927). In the detection of gelatine by the above method in sour cream, some soluble decomposition products may produce a precipitate; but this is insoluble in hot water whereas the gelatine precipitate is soluble. In such cases the mixture is shaken vigorously and filtered; the precipitate is washed first with ammonia water until the washings are alkaline to litmus, and then with water; the washed precipitate is heated to boiling with 10 to 20 c.c. of water; filtered and cooled; and the filtrate is treated with an equal volume of picric acid solution; in the presence of gelatine a precipitate is formed. By this method it is possible to detect as little as 0·5 per cent. of gelatine.

Test for starch. A few c.c. of the cream are diluted with water in a porcelain dish and a few drops of iodine solution added; a blue colour is obtained when starch is present.

Viscogen in cream. Viscogen is a solution of cane sugar in lime, a very small quantity of which has a remarkable effect in thickening either milk or cream. Its presence may be detected either by the determination of the relative proportion of lime in the ash or by the determination of the alkalinity of the ash.

A small proportion of viscogen increases the alkalinity of the ash very materially.

Test for viscogen. Mix 1 c.c. of sesamé oil ; 1 c.c. of hydrochloric acid and 0·5 gram of the suspected cream ; shake the mixture thoroughly for one minute and allow it to stand for 30 minutes ; in the presence of viscogen a crimson colour is produced. As little as 1 per cent. of cane sugar can be detected by this method ; but for so small a quantity it is necessary to allow the mixture to stand for a somewhat longer period.

Preservatives in cream.

Boric acid. The most convenient method for the estimation of boric acid in milk and cream is that devised by Richardson (*Analyst*, 1913, vol. XXXVIII, p. 140).

Solution required : Copper sulphate solution containing 5 grams of copper sulphate per 100 c.c. 10 grams of cream are mixed with 40 c.c. of water and 5 c.c. of the copper sulphate solution are added, the mixture is well stirred and heated to boiling point for a few seconds ; about 0·5 gram of zinc dust is added, to remove the excess of copper ; the mixture is filtered through a dry filter paper and the contents of the paper are well washed ; to the cold filtrate add a few drops of phenol phthalein solution and N/10 sodium hydroxide until a permanent pink colour just appears ; glycerine is now added to the extent of one-third of the total volume and the mixture is again titrated with N/10 sodium hydroxide until the pink colour becomes permanent. Each c.c. of N/10 sodium hydroxide solution used after the addition of glycerine = 0·0062 gram of boric acid.

Fluorides. For the detection of fluorides, 100 grams of the cream are made alkaline with ammonium carbonate and boiled ; a few c.c. of calcium chloride solution are added and the boiling continued for five minutes ; the precipitate is collected on a filter paper ; washed, dried, and ignited in a platinum dish ; a few drops of concentrated sulphuric acid are added to the residue in the dish, which is then covered with a watch glass, partly protected by means of vaseline ; the dish is kept at 75°

to 85° C. for an hour, when in the presence of fluorides the watch glass will be found to have been etched.

Detection of hydrogen peroxide in cream. Solution required: Schardinger's Reagent. Mix together 5 c.c. of a concentrated alcoholic solution of methylene blue, 5 c.c. of formaldehyde and 190 c.c. of water.

Adam (*Jour. Pharm. Chim.* 1906, vol. 273) has shown that Schardinger's Reagent is invariably decolourized by a cream which has never contained H_2O_2, whatever its age may be; but a cream, which has once been treated with H_2O_2, loses its power of decolourizing the reagent and only regains it after about five days, when bacterial decomposition has set in.

Another method for the detection of hydrogen peroxide in milk and cream is described by Wilkinson and Peters (*Zeit. Nahr. Genuss.* 1908, vol. XVI, p. 172). It consists in adding ten drops of a 2 per cent. solution of benzidine to 10 c.c. of the sample, and then a few drops of dilute acetic acid; a blue colour is produced in the presence of hydrogen peroxide.

Typical analyses of cream.

	Raw Cream	Thick Cream	Devonshire Cream
Water ..	67·93	37·62	33·76
Fat ..	24·44	58·77	59·79
Lactose ..	2·96	1·46	1·01
Proteid ..	4·04	1·83	4·97
Ash ..	0·63	0·32	0·47
	100·00	100·00	100·00

The following analyses of various creams (in percentages) were kindly supplied by Mr West Knights.

	(1)	(2)	(3)	(4)	(5)	(6)
Total solids	59·46	57·44	39·72	34·24	53·46	58·00
Non-fatty solids.. ..	6·99	5·06	9·60	6·47	6·75	5·00
Fat	52·47	52·38	33·12	27·77	46·71	53·00
Ash	·50	·48	·54	·50	·50	·30
Boric acid	·42	·40	none	none	·38	none

Nos. (3) and (4) are poor in fat.

CONDENSED MILK.

The sale of condensed milk has increased considerably of late years and it is now an article of food of considerable importance. It is usually prepared by evaporating milk under diminished pressure to about one-third of its original volume, and this is sometimes followed by the addition of cane sugar. The percentage of fat in a condensed milk prepared from whole milk should not be less than ten. The actual adulteration of condensed milk is very rare, but misrepresentation is very common. For example, condensed milk made from separated milk and containing 1·0 per cent. of fat or less is frequently sold as condensed milk whereas the purchaser expects to obtain condensed whole milk. There is also considerable misrepresentation as regards the proportion of water necessary to dilute the sample in order to obtain a liquid comparable with new milk.

Methods of Analysis. Weigh out 10 grams of the condensed milk ; dissolve it in water and make it up to 100 c.c.

Total solids. 10 c.c. of the 10 per cent. solution are placed in a platinum dish and the mixture is evaporated to dryness ; the residue is dried in a water oven and weighed ; the increase in weight represents the solids in 1 gram of the condensed milk.

Ash. The residue from the total solid determination is carefully incinerated over a Bunsen flame ; cooled and weighed ; the residue multiplied by 100 gives the percentage of ash.

Fat. The fat can be determined by any of the methods described under milk but, as in the analysis of cream, a definite weight of the condensed milk should be diluted with a definite amount of distilled water.

Leach's method. 5 grams of the condensed milk are weighed into a small beaker ; dissolved in 15 c.c. of water and carefully washed into a Leffman-Beam bottle ; the bottle is filled nearly to the neck with water and 4 c.c. of copper sulphate solution (containing 7 grams of copper sulphate in 100 c.c.) are added ; the contents of the bottle are thoroughly mixed and rotated in the centrifuge ; the proteids and fat sink to the

bottom; the supernatant liquid contains the sugar; this liquid is drawn off with a long-drawn-out pipette; the precipitated fat and proteids are washed twice with water and finally sufficient water is added to bring the contents of the bottle to 15 c.c.; the test is then continued as in the ordinary Leffman-Beam method for the estimation of fat in milk. The reading on the bottle multiplied by 3·11 gives the percentage of fat.

Proteids. For this determination 20 c.c. of the diluted milk are used; and the amount of proteids is estimated by the Kjeldahl method as described for milk (p. 85).

Milk sugar. Weigh out 10 grams of the condensed milk; dissolve it in water; add a few drops of citric acid and make up to 250 c.c.; filter through a dry filter paper. Place 25 c.c. of copper Fehling's solution, 25 c.c. of alkaline Fehling's solution (prepared before, p. 87) and 25 c.c. of distilled water in a beaker of 250 c.c. capacity; place it in a boiling water bath; after the beaker has stood in the water bath for five minutes, run in from a pipette 25 c.c. of the filtrate obtained above; allow the beaker to remain in the water bath for exactly 12 minutes; quickly filter the solution through a good filter paper; wash the precipitate with hot water until the filtrate gives no alkaline reaction; dry the precipitate in a water oven; incinerate in a platinum dish and weigh as CuO; from the weight of CuO thus obtained subtract the weight of ash in a blank experiment. The weight of CuO multiplied by 0·5758 = the weight in grams of anhydrous lactose in the 25 c.c. taken for the experiment.

Example: in an experiment carried out as described 0·253 gram of CuO were obtained, therefore

$$0{\cdot}253 \times 0{\cdot}5758 \times 100 = 14{\cdot}57 \text{ per cent. of lactose.}$$

Cane sugar. A rough approximation of the cane sugar may be obtained by subtracting the sum of the percentages of the fat, ash, proteid and milk sugar from the total solids.

The cane sugar is accurately determined by Fehling's method. To 50 c.c. of the filtrate obtained as described in the determination of milk sugar, add one gram of citric acid and boil for 15 minutes; allow the solution to cool; just neutralize

it with caustic soda and make it up to 200 c.c. ; the copper reduction is then determined as described above.

From the weight of CuO obtained subtract the weight of CuO due to lactose, and the remainder multiplied by 0·4091 gives the cane sugar.

Some analyses of Condensed Milk in percentages.

	Sweetened	Unsweetened	Skimmed
Total solid matter..	76·5	36·3	67·4
Fat	10·0	11·3	0·7
Ash	1·9	1·5	1·9
Proteid	8·3	9·0	10·0
Lactose	15·6	14·5	15·4
Cane sugar	40·7	—	39·4

Dr Coutts' Report to the Local Government Board on condensed milks (*Food Reports*, No. 15, 1911) contains a considerable number of analyses of various brands of condensed milk, and it gives a very complete bibliography of the literature of the subject.

CHAPTER III

BUTTER, MARGARINE—LARD, DRIPPING, SUET—CHEESE, CREAM CHEESE—EDIBLE OILS

BUTTER AND MARGARINE.

During the churning of milk, the fat corpuscles and part of the casein are made to adhere together and are removed as butter ; the water, the remainder of the casein and the mineral salts are left behind as buttermilk. Butter fat consists of the glycerides of butyric, caproic, capric, caprylic, myristic, palmitic, stearic and oleic acids. After milk, butter is probably the most adulterated of all articles of food ; the usual adulterations practised are complete or partial substitution of margarine and excess of water.

The amount of water in butter is controlled by the Sale of Butter Regulations, 1902, and by the Butter and Margarine Act,

1907, under which the maximum amount of water in butter may not exceed 16 per cent. Butter containing more than 16 per cent. of water may be legally sold, provided that it is sold under a fancy name approved by the Board of Agriculture.

Analysis of butter. Determination of water. Weigh out 5 grams of the sample in a flat bottomed platinum dish and place it in an air oven at 105° C. until, on imparting a circular motion to the dish no "crackling" is heard; the water will then have been driven off; the dish is allowed to cool in a desiccator and reweighed; the loss in weight multiplied by 20 gives per cent. of water.

Example: The loss in weight on 5 grams of a sample of butter was 0·631 gram, then $0{\cdot}631 \times 20 = 12{\cdot}62$ per cent. of water.

Salt. The residue from the water determination is incinerated over a Bunsen flame or in a small furnace at as low a temperature as possible, until a white or nearly white ash is obtained; this ash may for all practical purposes be regarded as salt. While there is no fixed standard for salt in butter, yet good salted butter contains from 0·5 to 6 per cent. of salt, and anything above 6 per cent. can only be regarded as an adulteration.

Curd. Weigh out 5 grams of the butter into a small beaker; dissolve the fat in petroleum ether and filter through a dried and weighed filter paper; well wash the residue on the paper with petroleum ether; to remove all fat, dry the paper in a water oven and reweigh; the increase in weight represents curd and ash; incinerate the filter paper and residue in a platinum dish; subtract the weight of the filter ash and deduct the remainder from the weight of the curd and ash, thereby giving the weight of curd.

Fat. The filtrate from the curd determination is placed in a weighed fat-flask and the petroleum ether is evaporated off; the flask is then dried in a water oven to constant weight; the increase in weight of the flask multiplied by 20 gives the percentage of fat.

Sorting test for butter samples. A rough method of sorting butter samples containing excess of water from those which have

a water content within the above mentioned limits has been proposed by Russell (*Jour. Roy. San. Inst.* 1906, vol. XXVII, p. 561). A glass cylinder graduated in c.c. and having a capacity of about 50 c.c. is filled with the butter sample and placed in a water oven until the butter is completely melted; the cylinder is then placed in a centrifugal machine and rotated for two minutes; after the rotation the curd and water will have settled to the bottom leaving a clear column of fat; the volume of the fat column is read off and reduced to a percentage of the total volume. Should the volume of the fat be less than 82 per cent., the percentage of water is estimated by the method given above. This method is exceedingly useful where a large number of butters have to be examined, as the saving of time is very large and the butter samples passed by the method invariably fall within the limits for water.

Examination of the fat. A portion of the sample is placed in a small beaker, in a water oven until melted; the fat rises to the top and is carefully decanted through a dry filter paper; the fat will pass through quite clear and bright.

Preliminary examination. A few years ago the Valenta Acetic-acid Number and the Zeiss Butyro-refractometer Number were sufficiently good sorting tests to indicate the presence of margarine in butter; but, since the introduction of cocoanut oil, most margarines give numbers closely corresponding to those for pure butter fat, thereby rendering these determinations useless.

Heat of bromination. For this determination, weigh out exactly 1 gram of butter fat into a vacuum jacketed test-tube; add 10 c.c. of chloroform from a pipette; mix well with a thermometer (graduated in $\frac{1}{5}$° C.); note the initial temperature of the mixture and surround the tube with cotton wool; now from a pipette allow 1 c.c. of bromine to run down the side of the tube, without stirring; and note the maximum temperature reached; the increase in temperature is the heat of bromination.

The heat of bromination of a genuine butter should be from 4 to 7; of an old fashioned margarine from 9 to 10; of a cocoanut oil from 1·5 to 1·7. The heat of bromination multiplied by 5·5 gives the iodine absorption number.

Determination of the Reichert-Wollny number. The Reichert-Wollny number is a measure of the volatile fatty acids in the sample in terms of N/10 alkali. It is most conveniently determined by Leffman and Beam's modification with glycerol-soda solution; this method has a great advantage over the older and more tedious alcoholic method.

Solutions required: Glycerol-soda solution. Weigh out 100 grams of sodium hydroxide; dissolve it in 100 c.c. of distilled water and allow the solution to stand until clear.

Measure out 20 c.c. of the clear solution and 100 c.c. of pure glycerine; mix the two; keep the mixture in a capped bottle fitted with a rubber bung.

Sulphuric acid solution containing 20 c.c. of concentrated sulphuric acid in 100 c.c.

N/10 sodium hydroxide solution.

The process. Weigh out into a 300 c.c. Jena flask, 5 grams of the clear butter fat; add 20 c.c. of the glycerol-soda solution; carefully heat the flask over a Bunsen burner; the mixture may froth considerably at first, but this ceases as soon as all the water has been driven off; the heating is continued until the mixture becomes clear, when complete saponification will have taken place; 135 c.c. of hot water are now carefully added at first drop by drop, the flask being shaken to avoid frothing; the soap formed dissolves in the water, and, as soon as the solution is complete, 5 c.c. of the sulphuric acid solution are added, and a small piece of pumice to prevent bumping; the flask is then connected to a special condenser and heated with a naked flame until 110 c.c. have been distilled; this operation should occupy about 30 minutes. The distillate is well mixed and filtered through a dry filter paper; 100 c.c. of the filtrate are placed in a conical flask; two or three drops of phenol phthalein solution are added, and then N/10 sodium hydroxide run in from a burette until a permanent pink colour is obtained; the number of c.c. of N/10 sodium hydroxide solution used multiplied by 1·1 gives the Reichert-Wollny number.

Genuine butter fat should not give a Reichert-Wollny number of less than 24; the Reichert-Wollny number of margarine is from 0·2 to 1; and of cocoanut oil from 6·5 to 7·5.

The Shrewsbury and Knapp number (*Analyst*, 1910, vol. xxxv, p. 385). The Reichert-Wollny process is not capable of detecting small quantities of cocoanut oil; and Shrewsbury and Knapp have devised a process by which they claim to be able to detect as little as 5 per cent. of cocoanut oil in butter fat.

Solutions required: Glycerol-soda solution. Approximately 10N. sodium hydroxide solution is prepared, and 100 c.c. of this solution are mixed with 500 c.c. of glycerine.

Dilute sulphuric acid solution. 100 c.c. of concentrated sulphuric acid are mixed with 400 c.c. of distilled water.

Alcohol. Industrial methylated spirit containing 90 per cent. of absolute alcohol by weight.

The process. 5 grams of the filtered fat are weighed into a 300 c.c. Jena flask and saponified with 20 c.c. of the glycerol-soda solution; the soap obtained is at once carefully diluted with boiling water and washed into a separator; the total quantity of boiling water used is exactly 200 c.c.; 5 c.c. of the dilute sulphuric acid solution are at once added and the mixture is vigorously shaken for one minute and allowed to stand for five minutes; the water is then run off from the insoluble fatty acids. The fatty acids in the separator are now dissolved in 50 c.c. of the alcohol, and the solution is poured into a flask and heated, with a piece of pumice to prevent bumping; 30 c.c. of cold water are run into the separator; and immediately the alcohol solution in the flask boils, it is poured into the water in the separator; the mixture is poured back into the flask, which is rotated, and then back into the separator; it is then shaken for 30 seconds and allowed to stand for three minutes so that the insoluble fatty acids may separate; 70 c.c. of the alcoholic solution are run off and titrated with N/10 sodium hydroxide solution using phenol phthalein solution as an indicator.

Average numbers obtained are:

Butter	28 c.c.
Margarine (free from cocoanut oil) ..	20 c.c.
Cocoanut oil	163 c.c.

Determination of fat in butter (A. Hesse, *Zeit. Nahr. Genuss.* 1904, 673). 2 grams of the sample are weighed into a stoppered cylinder and melted by the addition of 8 c.c. of boiling water; 1 c.c. of ammonia and 10 c.c. of alcohol are added and the cylinder is shaken until the proteids are dissolved; allow the mixture to cool; add 25 c.c. of ether, followed by 25 c.c. of petroleum ether; the whole is then well shaken and allowed to separate; the ether layer is then pipetted off and the residue again shaken with 25 c.c. of ether, which is pipetted off; and finally with 25 c.c. of ether and 25 c.c. of petroleum ether. The mixed washings are evaporated and the residue dried in a water oven and weighed; the weight of residue obtained multiplied by 50 gives the percentage of fat.

Preservatives in butter. The most common preservatives used in butter are borax and boric acid or a mixture of these two. About 20 per cent. of the butter sold in this country contains this preservative.

The Report of the Departmental Committee on the use of preservatives and colouring matters of foods (1901, Cd. 833) recommended that the only preservatives to be allowed in butter should be boric acid or borax, or mixtures of these two in proportions not exceeding 0·5 per cent. calculated as boric acid. At the same time it is very doubtful whether the use of this substance is necessary and certainly 0·5 per cent. is excessive.

Determination of the boric acid in butter. Weigh out 10 grams of the well mixed sample into a 300 c.c. flask and add 200 c.c. of water; warm the mixture over a Bunsen burner until the fat is melted; shake the mixture well and allow it to stand until the fat rises to the surface; siphon off the liquid into a measuring cylinder and note the volume. Place the liquid in a conical flask; add a few drops of methyl orange and dilute sulphuric acid until a pink colour is obtained; boil the solution for two or three minutes; cool in water; and, when cold, add a few drops of phenol phthalein solution, followed by N/10 sodium hydroxide solution until a faint pink colour appears; to the mixture is then added one-third of its volume of glycerine and N/10 sodium hydroxide solution until a permanent pink

colour is obtained. The number of c.c. of N/10 sodium hydroxide solution used after the addition of glycerine multiplied by 0·0062 gives the amount of boron compounds calculated as boric acid in 10 grams of the butter.

Typical Analyses of Butter (in percentages).

	Irish	English	"Milk Blended"
Fat	81·7	91·5	74·2
Curd	2·3	1·7	1·1
Salt	1·8	—	2·6
Water	14·2	6·8	22·1

The following analyses of butters have been kindly supplied by Mr West Knights.

	(1)	(2)	(3)	(4)	(5)	(6)
Water (per cent.) ..	9·27	12·97	14·57	10·23	15·67	9·43
Reichert-Wollny number in c.c. of N/10 NaHO ..	30·2	24·0	4·00	28·0	3·8	2·0
Boric acid (per cent.) ..	none	·28	·12	none	·25	·20

Nos. (1), (2) and (4) are genuine butters; nos. (3), (5) and (6) consist entirely of margarine sold as butter.

Margarine must not contain more than 10 per cent. of added butter, 16 per cent. of water, and 0·5 per cent. of boric acid.

LARD.

Lard consists of the mixed fats obtained from various parts of the hog; the best lard being obtained from the membranes surrounding the kidneys and from the back.

The chief adulterants of lard are cottonseed oil, beef stearine, paraffin wax and cocoanut oil.

The presence of cocoanut oil is detected, and its amount estimated by Shrewsbury and Knapp's method as described under butter (p. 108).

Detection of cottonseed oil. The presence of cottonseed oil in lard is detected by Halphen's test.

Solutions required: A solution of sulphur in carbon disulphide, containing 1 gram of sulphur in 100 c.c. of carbon disulphide; this solution is mixed with an equal volume of fusel oil.

Equal volumes of the oil and reagent, about 5 c.c., are placed in a test-tube, loosely stoppered with cotton wool, and heated

in a boiling brine bath for 15 minutes. In the presence of cottonseed oil a deep orange to red colour is obtained. Should a very faint colour be obtained it does not necessarily indicate the presence of added cottonseed oil, as the fat obtained from hogs fed on cottonseed meal or cake may give a faint reaction.

Determination of the iodine absorption. *Solutions required:* Wij's iodine solution (*Ber. Chem. Ges.* 1898, vol. xxxi, p. 750). Weigh out 13 grams of pure iodine and dissolve it in 1000 c.c. of glacial acetic acid (99 per cent.); determine the strength of the iodine solution with a standard solution of sodium thiosulphate; a slow stream of chlorine gas, free from hydrochloric acid, is now passed through the solution until the volume of the standard solution of sodium thiosulphate used for the titration is doubled; this occurs when the original dark brown of the solution is changed to light yellow.

A solution of potassium iodide containing 10 grams of the salt in 100 c.c. of water.

N/10 sodium thiosulphate solution.

The process. Weigh out 1 gram of the oil or fat; dissolve it in chloroform, and make the solution up to 100 c.c. with chloroform. Into a wide-mouthed stoppered glass bottle, measure out 20 c.c. of the chloroform solution of the fat; add 20 c.c. of the Wij's iodine solution and set aside for 20 minutes. At the end of 20 minutes, 25 c.c. of the potassium iodide solution are added; the whole is then diluted with 300 c.c. of water and the N/10 sodium thiosulphate solution is run in from a burette, with vigorous agitation of the bottle, until the colour is nearly discharged; a few c.c. of dilute starch solution are then added and the titration continued until the blue colour is discharged. A blank experiment should be performed upon the materials used.

The iodine absorption is obtained from the formula

$$\frac{T \times 0{\cdot}0127 \times 100}{W}$$

where T = the difference in c.c. in the volume of sodium thiosulphate solution used for the blank experiment and for the oil.

W = the weight of oil taken.

The iodine absorption number for lard is from 50 to 63 ; for beef stearine from 34 to 47 ; and for cottonseed oil from 101 to 116.

Example : In an experiment with lard 1 gram of the fat was dissolved in 100 c.c. of chloroform and 20 c.c. of the solution used for the experiment.

The volume of sodium thiosulphate solution used for the blank experiment was 39·7 and for the lard 30·3. Then the iodine number of the sample of lard was

$$\frac{9{\cdot}4 \times 0{\cdot}0127 \times 100}{0{\cdot}2} = 59{\cdot}69.$$

Test for paraffin wax (Holde's method, *Jour. Soc. Chem. Ind.* 1892, p. 637). Ten drops of the melted lard are saponified in a test-tube with 5 c.c. of approximately N/2 alcoholic potash ; to the clear soap solution, water is added in quantities of 1 c.c. at a time, the solution being observed after each addition. Pure lard gives a clear solution after dilution with 5 c.c. of water, and as little as 0·5 per cent. of paraffin wax gives a characteristic turbidity.

Detection of beef stearine. The detection of beef stearine in lard is exceedingly difficult. In the absence of other adulterants, it may be detected by the iodine absorption but it is possible by judiciously mixing cottonseed oil and beef stearine to prepare a product giving an iodine number within the limits for pure lard.

Belfield (*Analyst*, 1898, vol. XIII, p. 70) proposed a microscopical method in which 3 grams of the sample are dissolved in 15 c.c. of warm ether in a test-tube loosely plugged with cotton wool ; the solution is cooled to 13° C. and allowed to stand at that temperature overnight ; the crystals obtained are examined microscopically. Lard crystallizes in flat rhomboidal plates, while in the presence of beef stearine the characteristic tufts of cylindrical needles are obtained.

Water. Lard is occasionally, though not frequently, adulterated by the addition of water. Pure lard should be absolutely free from water, and also from ash.

DRIPPING AND SUET.

Dripping and suet are the rendered fats from various animals, chiefly cows, sheep and hogs. They often contain water as an adulterant, from which they should naturally be free. The addition of starch (in the form of oatmeal or corn-flour) to suet to prevent greasy particles from adhering and coalescing together is becoming common; but this addition must be regarded as an adulteration if the sample is sold without adequate declaration.

The fat should be examined by the Shrewsbury and Knapp process (p. 108) and the iodine absorption (p. 111) in order to detect the presence of vegetable oil.

CHEESE.

Cheese is the compressed and "ripened" product obtained from milk which has been either naturally or artificially soured. The "ripening" process is essentially putrefactive and requires several months to develop the characteristic flavour. Cheese consists principally of fat, nitrogenous bodies, water, lactose and mineral matter. It is a noteworthy fact that there is no standard for cheese in this country, and as a result cheese is sold made from either whole or skimmed milk without notification to the purchaser. It often contains only minute traces of fat; whereas it is an offence to sell a cheese containing margarine fat without notification. The varieties of cheese take their names from the town or district in which they are made; for example, Cheshire and Cheddar.

Analysis of cheese. Fat. The fat in cheese is best determined by Ratzlaff's method (*Milch. Zeitung*, 1903, vol. v); 6 grams of the cheese are weighed into a small Erlenmeyer flask about 4 cm. in diameter; 10 c.c. of concentrated hydrochloric acid are added; a small funnel is placed in the neck of the flask and the contents are heated on a water bath until they are dissolved. The flask is now placed on a sand bath; carefully heated to boiling and kept boiling for three minutes. The flask is then

allowed to cool; 5 c.c. of ether are added and the contents poured into a Stokes tube, the flask is rinsed out once with 5 c.c. of ether and twice with 5 c.c. of petroleum ether; the tube is filled to the upper mark with a mixture of equal parts of ether and petroleum ether and closed with a well washed cork. The contents of the tube are thoroughly mixed and allowed to separate; the volume of the ether layer is then read off and 20 c.c. are pipetted out into a dried and weighed fat-flask; the ether is evaporated off and the residual fat is dried in the water oven for 45 minutes and weighed. The mixture of ether and petroleum ether dissolves the fat only and no other constituent of the cheese.

Example: 5 grams of cheese were taken for the estimation; the volume of the ether layer was 25·5 c.c., the weight of fat obtained from 20 c.c. was 1·976 grams; then the percentage of fat in the cheese was

$$\frac{1{\cdot}967 \times 25{\cdot}5 \times 100}{20 \times 5} = 50{\cdot}39.$$

Water. A portion of the sample is cut into very thin slices and 5 grams are weighed into a platinum dish and dried in an air oven at 105° C. to constant weight; the loss in weight is reckoned as water.

Ash. The dry residue obtained from the water determination is carefully incinerated at a low red heat; the residue is weighed and calculated to a percentage.

Proteids. The proportion of proteids is obtained by multiplying the percentage of nitrogen, as determined by the Kjeldahl process described under milk (p. 85), by 6·38.

Test for starch. A small portion of the sample, from which the fat has been extracted, is boiled with water and filtered; to the filtrate a few drops of a dilute iodine solution are added; when a blue colour is obtained in the presence of starch.

Examination of the fat. It is of course necessary to examine the fat for the presence of other fats than butter fat; and, in order to obtain sufficient fat for this purpose, about 50 grams of the sample are chopped up into small pieces and placed in a muslin bag, which is suspended over a beaker by means of

a glass rod ; the whole is then placed in a water oven, when the fat will melt out quite clear and should be examined by the Reichert-Wollny process (p. 107).

CREAM CHEESE.

Cream cheese is the product obtained from the souring of milk either with or without rennet, and, according to Moor (*Suggested Standards for Foods and Drugs*, p. 88), should contain at least 40 per cent. of fat. According to Cribb (*Analyst*, 1909, vol. XXXIV, p. 45), in a genuine cream cheese the ratio $\frac{\text{fat}}{\text{protein}}$ should exceed 1 (protein = nitrogen number multiplied by 6·37, see p. 86). Numbers of cream cheeses are on the market containing only a very small proportion of fat and obviously made from machine-skimmed milk. The sale of these substances as cream cheese without notification can only be regarded as a fraud. According to the standard of the United States of America, cream cheese is a cheese made from milk or cream, or from milk containing not less than 6 per cent. of fat.

The analysis of a cream cheese is carried out precisely as if it were an ordinary cheese.

Typical Analyses of Cheese (in percentages).

	Fat	Water	Casein, etc.	Ash
Cheddar, English ..	31·57	35·60	28·61	4·32
Cheddar, American ..	37·09	30·09	29·25	3·57
Cheshire	30·68	37·11	27·79	4·42
Dutch	22·78	41·30	28·82	7·10
Gorgonzola	34·34	31·85	29·23	4·58
Roquefort	34·38	32·26	28·48	4·88
Stilton	39·13	23·57	33·79	3·15
Lard	24·66	31·30	38·87	5·17
Margarine	28·80	30·95	36·27	3·98
Cream	67·15	23·99	8·17	0·69
Cream	47·04	36·61	15·44	0·81
Cream	1·16	70·66	25·37	2·81

The last sample of cream cheese has been made from a machine-skimmed milk.

EDIBLE OILS.

Olive oil. Olive oil is the colourless to yellow oil obtained by extraction or compression from the fruit of *Olea sativa* and consists chiefly of olein and palmitin. Owing to its high price olive oil is very extensively adulterated, the chief adulterant being Arachis oil.

Test for Arachis oil in olive oil. The sample is saponified with an equal weight of alcoholic potassium hydroxide solution (200 grams of solid potassium hydroxide in 500 grams of 90 per cent. alcohol) on the water bath for 30 to 40 minutes and allowed to cool down to a temperature between 0° and 6° C. In the presence of as little as 5 per cent. of Arachis oil granular masses of potassium arachidate separate, on the walls of the test-tube, which are insoluble in alcohol. Large quantities of Arachis oil are indicated by the solidification of the whole mass.

Estimation of Arachis oil. *Solutions required*:

Alcoholic potash solution containing 85 grams of solid potassium hydroxide in 1000 c.c.

Acetic acid solution, 1·5 c.c. of which exactly neutralize 5 c.c. of the alcoholic potash solution.

Alcoholic hydrochloric acid solution containing 1 per cent. of hydrochloric acid in 70 per cent. alcohol.

The process. Saponify 5 grams of the oil with 25 c.c. of the alcoholic potash solution; exactly neutralize the potash with 7·5 c.c. of the acetic acid solution and rapidly cool the vessel by immersing it in water. At the end of one hour, the precipitate is collected on a filter paper and washed with 70 per cent. alcohol, containing 1 per cent. hydrochloric acid, the temperature being maintained between 15 and 20° C.; the washing is continued until the filtrate does not become turbid on the addition of water. The precipitate, which contains other acids besides arachidic, is dissolved off the filter paper with 25 to 50 c.c. of boiling alcohol (92 to 93 per cent.); from 8 to 16 c.c. of water are added to the solution, in order to reduce the strength of the alcohol to about 20 per cent., and the liquid is cooled for one hour in water at a temperature below 20° C.

The precipitate, which consists of arachidic acid, is collected on a dried and weighed filter paper, washed with 70 per cent. alcohol (free from hydrochloric acid) until the filtrate remains clear on the addition of water, dried at 100° C. and weighed.

Arachis oil contains 4 to 5 per cent. of arachidic acid, consequently the amount of arachidic acid found must be multiplied by 20 to obtain the amount of Arachis oil present.

Sesamé oil. This oil is the yellow odourless product obtained from *Sesamum orientale* and *Sesamum indicum* and consists principally of olein, stearin, palmitin and linolin. In some countries the addition of sesamé oil to margarine is compulsory in order to facilitate the detection of that fat.

Baudouin's test for sesamé oil (*Zeit. Ang. Chem.* 1892, pp. 509–510). *Solution required:* A hydrochloric acid solution of cane sugar, containing 1 gram of cane sugar in 100 c.c. of hydrochloric acid (specific gravity 1·2).

20 c.c. of the oil are mixed with 10 c.c. of the cane sugar solution, the mixture is well shaken and allowed to stand. As little as 2 per cent. of sesamé oil imparts a crimson colour to the aqueous layer.

Cottonseed oil. Cottonseed oil is obtained from the seeds of *Gossypium*, and consists principally of olein, stearin, palmitin and linolin. Apart from its legitimate use, it is chiefly used as an adulterant of other oils and fats.

Becchi's reaction for cottonseed oil (*Analyst*, 1887, XII, 170).

Solutions required: A solution containing

Silver nitrate	2 grams,
Alcohol	200 c.c.,
Ether	40 c.c.,
Nitric acid	0·1 c.c.

6 c.c. of the oil are mixed with 3 c.c. of the above solution in a test-tube and immersed in a boiling water bath for 15 minutes; in the presence of cottonseed oil a more or less strong reduction of silver is obtained. Halphen's test for cottonseed oil has been described under lard (p. 110).

Castor oil. This oil is the colourless transparent oil obtained from castor seeds (*Ricinus communis*) and consists of tristearin, dihydroxistearin and ricinolin. Pure castor oil is miscible with absolute alcohol in all proportions and is practically insoluble in petroleum ether.

CHAPTER IV

TEA—COFFEE—CHICORY—COCOA

TEA.

Tea consists of the leaves and leaf buds of species of *Thea*, prepared by fermenting or drying and firing. Tea has been used in China for 6000 years; it was first introduced into Europe by the Portuguese in the middle of the 16th century, and in 1912 it was imported into the United Kingdom to the extent of 361 millions of pounds, 300 millions of which came from India and Ceylon.

There are two chief varieties of tea, green and black; the difference between them is solely in the method of manufacture. These varieties are subdivided, green teas into gunpowder, hyson and imperial; black teas into Bohea, Congou, Pekoe and Souchong. Caper tea consists of tea dust made into small hard balls with gum and polished with graphite. The adulteration of tea in this country has practically ceased to exist; 491 samples of tea were examined under the Sale of Foods and Drugs Acts in 1911 and all were found to be genuine. This is due to the fact that all tea imported into the United Kingdom is examined by the Customs Authorities acting under the provisions of the Sale of Foods and Drugs Acts and the consequent prohibition of the importation of adulterated tea. In the past tea was adulterated by the addition of exhausted leaves, foreign leaves, mineral matter or foreign astringent matter.

Examination of tea. The complete analysis of tea is very

difficult owing to its complex composition and a partial analysis is generally undertaken.

Detection of foreign leaves. A portion of the sample is soaked for a short time in hot water; the leaves opened out and examined with a hand glass or a low power of a microscope, when the presence of foreign leaves is easily detected, genuine tea leaves being lanceolate in form.

Detection of foreign astringent matter. 1 gram of genuine tea and 1 gram of the suspected sample are extracted with separate portions, of 100 c.c. each, of boiling water; the solutions are strained and a slight excess of lead acetate added; the solutions are then filtered, and to 20 c.c. of each of the filtrates, a few drops of silver nitrate solution are added and the whole carefully warmed; pure tea gives a grey precipitate, but tea containing catechu gives a large brown precipitate of reduced silver. It is claimed that in this way as little as 2 per cent. of catechu may be detected.

Determination of ash. 5 grams of the tea are weighed out into a platinum dish and carefully incinerated over a Bunsen flame until a white or greyish white residue is obtained; the weight of the residue multiplied by 20 gives the percentage of ash.

Determination of the insoluble ash and silica. The total ash as obtained above is extracted with water and filtered, and the residue well washed with hot water; the filtrate is set aside for the alkalinity determination; the residue on the filter paper is dried in a water oven, incinerated and weighed; from this weight the weight of the ash of the filter paper is subtracted, the difference is the insoluble ash; this insoluble ash is then extracted with dilute hydrochloric acid and filtered; the filter paper is well washed with hot water, dried in a water oven and incinerated; the residue, less the weight of the ash of the filter paper, is silica.

Caper tea is very liable to contain a comparatively large amount of silica but any percentage above three must be regarded as an adulteration. The soluble ash is obtained by subtracting the insoluble from the total ash; or it may be obtained directly by evaporating the filtrate from the insoluble

ash to dryness on the water bath and heating the residue in the water oven till constant ; when the weight of residue obtained multiplied by 20 gives the percentage of soluble ash.

Determination of the alkalinity of the ash. The filtrate from the insoluble ash determination is treated with N/10 sulphuric acid, using methyl orange as an indicator, until the solution has a faint pink colour, when the number of c.c. of N/10 sulphuric acid used multiplied by 0·0047 gives the amount of alkalinity, calculated as K_2O on 5 grams of the tea.

Example: The filtrate from the ash of 5 grams of tea required 14·5 c.c. of N/10 sulphuric acid to produce a permanent pink colour, then $14{\cdot}5 \times 0{\cdot}0047 \times 20 = 1{\cdot}36$ per cent. of alkalinity calculated as K_2O.

Estimation of exhausted leaves. The percentage of exhausted leaves in a sample of tea is calculated either from the soluble ash or from the alkalinity. The soluble ash of a genuine tea should not be less than 3 per cent. ; and the alkalinity should not be less than 1·3 per cent. calculated as K_2O.

Example: A sample of tea gave 2·13 per cent. of soluble ash, then the percentage of exhausted leaves in the sample is $100 - \frac{2{\cdot}13 \times 100}{3} = 29$ per cent. Another sample of tea gave an alkalinity of 0·85 per cent. calculated as K_2O, then the percentage of exhausted leaves in the sample is

$$100 - \frac{0{\cdot}85 \times 100}{1{\cdot}3} = 34{\cdot}6 \text{ per cent.}$$

Typical Analyses of Tea (in percentages).

	(1)	(2)	(3)	(4)	(5)
Total ash	5·50	4·32	4·92	13·47	6·38
Soluble ash	4·06	0·46	2·12	2·80	3·21
Insoluble ash	1·37	3·58	2·55	4·41	2·49
Silica	0·07	0·28	0·25	6·26	0·68
Alkalinity (as K_2O) ..	1·67	0·28	0·92	—	1·79

No. (1) is a genuine tea · No. (2) is a sample of exhausted leaves ; No. (3) is a sample of tea containing 30 per cent. of exhausted leaves ; No. (4) is a sample of adulterated caper tea ; while No. (5) is a genuine caper tea.

The following analyses of samples of tea have been kindly supplied by Mr West Knights. All these are genuine. The microscopic examination was satisfactory in each sample.

	(1)	(2)	(3)	(4)	(5)	(6)
Soluble ash	3·90	3·20	4·10	3·20	3·75	4·20
Insoluble ash	1·45	2·47	1·75	2·60	2·10	1·90
Total ash	5·35	5·67	5·85	5·80	5·85	6·10

Typical Analysis of the Ash of Tea.

Potash (as K_2O)	30·04	Magnesia (as MgO)	4·87
Sand (as SiO_2)	17·78	Alumina (as Al_2O_3)	3·42
Phosphate (as P_2O_5)	14·68	Oxide of manganese (as Mn_3O_4)	1·37
Carbonate (as CO_2)	9·58	Soda (as Na_2O)	1·07
Lime (as CaO)	8·74	Chlorine (as Cl)	1·07
Sulphate (as SO_3)	6·54	Iron (as Fe_2O_3)	0·84
			100·00

COFFEE.

Coffee is the seed of *Coffea arabica* or *Coffea liberica*, roasted and ground or otherwise prepared in a form suitable for a decoction. *Coffea arabica* is indigenous to N.E. Africa and Arabia. Coffee, as a beverage, has been known in Abyssinia from time immemorial, and was introduced into England in the middle of the 17th century. The coffee used in this country consists chiefly of five varieties, Mocha, Jamaican, Indian, Liberian and Brazilian.

As soon as the ripe fruit has been gathered, it is dried and freed from the husk ; the raw beans are afterwards roasted in gauze cylinders over a gas or coal fire at a maximum temperature of 200° C. ; during this process, the bean loses about 20 per cent. of its weight ; the roasted bean is then ground for preparing the infusion.

Coffee has been adulterated by facing with ochre, prussian blue, etc. in order to improve its appearance ; but these are easily detected by the increased percentage of ash, which in a genuine coffee should fall between 3·5 and 4 per cent. A number of imitation coffees have been placed on the market, among which may be mentioned black malt, from which products, having the general appearance of coffee beans, are prepared by mixing with gum and moulding. The most

common adulterant is chicory, this is the dried and roasted root of *Chicorium intybus*, and its influence on a coffee mixture has been summed up by Winter Blyth (*Foods: their Composition and Analysis*, p. 359) as follows : (1) it increases the gum, (2) it increases the sugar, (3) it decreases the fatty matter, (4) it decreases the tannin, (5) it modifies the constituents of the ash.

Detection of chicory. The detection of chicory is a comparatively simple matter. A few grams of the sample are placed in a beaker and thoroughly extracted with boiling water ; the mixture is kept boiling for about three minutes with constant stirring, and afterwards allowed to cool and settle ; as soon as the grains have settled to the bottom of the beaker, the water layer is carefully poured off ; if no chicory is present the grains will remain at the bottom of the beaker, but, in the presence of chicory, some of the grains will rise up the sides of the beaker, and these grains should be collected and examined under the microscope, when the spiral vessels of the chicory root will be easily recognized. The blunt ends of the chicory cells are quite distinct from the lanceolate shape of the coffee cells.

Estimation of chicory. It has been found that the specific gravity of a 10 per cent. decoction of chicory is much higher than that of a similar decoction of coffee and from this the percentage of chicory can be determined. 10 grams of the sample are weighed out into a 200 c.c. beaker and 100 c.c. of water are added ; the whole is then heated over a Bunsen flame to boiling and kept at the boil, with constant stirring for three minutes ; the mixture is allowed to settle and the decoction poured off through a filter paper into a 100 c.c. flask ; the grounds are washed with a very small quantity of hot water, which is also poured through the filter paper ; the filtrate is now cooled to 60° F. and made up to 100 c.c. ; the specific gravity of the decoction is taken either with a Westphal balance or with a specific gravity bottle, and the amount of chicory calculated from this.

Example: The specific gravity of a 10 per cent. decoction of a sample was 1020. The specific gravity of a 10 per cent.

decoction of coffee should not exceed 1009·5, that of chicory should not be less than 1024·5, then

$$\frac{(1020 - 1009{\cdot}5) \times 100}{1024{\cdot}5 - 1009{\cdot}5} = 70 \text{ per cent. of chicory.}$$

The above method is not very accurate as the results obtained are influenced by the fineness of the coffee, by the method of working, and also by the variety of the coffee. Since the specific gravity of the 10 per cent. decoction is not constant, this difficulty has been overcome by a method devised by E. W. T. Jones (*Analyst's Laboratory Companion*, p. 133), which gives much more accurate results.

About 10 grams of the sample are placed in the water oven till dry; 5 grams of the dry sample are weighed out into a 300 c.c. beaker; about 200 c.c. of water are added, and the whole boiled for 15 minutes; the mixture is then allowed to settle and the liquid is strained through a wire gauze into a 250 c.c. graduated flask; the grounds are now boiled for five minutes with 50 c.c. of water and the liquid strained off as before; the mixed filtrates are then cooled, made up to the mark and filtered through a dry filter paper; 50 c.c. of the filtrate are measured out into a weighed platinum dish and evaporated to dryness on a water bath and finally dried in the water oven; the residue is calculated as a percentage extract on the dry sample.

Coffee when treated in this way gives a remarkably constant extract of 24 per cent., while the average extract of chicory is 70 per cent. The percentage of chicory in the sample is calculated from the formula

$$X = \frac{E - 24}{0{\cdot}46}$$

where X = the per cent. of chicory,
E = the per cent. of extract found.

Example: A suspected sample gave a percentage extract of 47, then $\frac{47 - 24}{0{\cdot}46} = 50$ per cent. of chicory.

Of late years a practice has sprung up of selling mixtures of coffee and chicory, which are labelled "coffee" in very large

letters and "with chicory" in very small letters; in some cases these mixtures contain very large proportions of chicory, sometimes upwards of 75 per cent.; in one case, in the City of Bristol, a sample was purchased containing at least 95 per cent. of chicory. There is no doubt that mixtures should not be legally sold containing more than 50 per cent. of chicory, as it has been decided by the High Court of Justice (Liddiard *v.* Reece and Horder *v.* Meddings, 44 J.P. 233 and 234) that where in the opinion of the bench the excess of chicory is added for the purpose of fraudulently increasing the bulk, an offence is committed. This difficulty is surmounted by the standard in the State of Victoria, where a mixture labelled "coffee and chicory" must contain at least 50 per cent. of coffee, and one labelled "chicory and coffee" must contain at least 50 per cent. of chicory.

The following analyses of various samples of coffee and coffee mixtures have been kindly supplied by Mr West Knights.

	(1)	(2)	(3)	(4)	(5)	(6)
Ash (per cent.) ..	4·40	5·10	4·10	3·90	5·40	5·53
Specific gravity of a 10 per cent. decoction ..	1009·3	1019·0	1009·0	1009·5	1017·0	1018·0
Chicory (per cent.) ..	none	59·0	none	none	47·0	53·0
Microscopic examination	satisfactory	showed cells of chicory	satisfactory	satisfactory	showed cells of chicory	showed cells of chicory

Analyses of the Ash of Coffee and Chicory.

	Coffee	Chicory
Potash (as K_2O)	53·72	25·80
Lime (as CaO)	6·06	10·61
Magnesia (as MgO) ..	8·34	7·03
Iron (as Fe_2O_3)	0·44	2·71
Sulphate (as SO_3) ..	3·05	10·83
Chlorine (as Cl)	0·72	4·23
Carbonate (as CO_2) ..	16·54	3·21
Phosphate (as P_2O_5) ..	11·13	10·59
Silica (as SiO_2)	—	10·09
Soda (as Na_2O)	—	14·90
	100·00	100·00

COCOA.

The cocoa of commerce is prepared from the seeds of *Theobroma cacao*; the seeds are contained in a pod and when first picked are white, but gradually darken on exposure to air; after removal from the pod the seeds are sun-dried and roasted; a process which develops the aroma and flavour; they are then cracked mechanically and the husks removed; the residue being known as cocoa nib. The seeds contain from 7 to 18 per cent. of husk which is used for adulterating the cheaper forms of cocoa and also as a useful addition to cattle food.

The following definitions of cocoa are of value: cocoa beans are the seeds of *Theobroma cacao*; cocoa of commerce consists of the powdered beans, more or less deprived of their fat; soluble cocoa or cocoa essence is the product obtained by treating powdered cocoa beans, more or less deprived of their fat, with alkali or an alkaline salt and should not contain more than 3 per cent. of alkali or alkaline salt, calculated as K_2CO_3. Prepared or compound cocoa is cocoa mixed with other substances such as flour or sugar or both.

The chief adulteration of cocoa is the substitution of prepared cocoa, containing sugar or starch or both for powdered cocoa.

Analysis of Cocoa.

Determination of the cold water extract. Thoroughly mix 5 grams of the cocoa with water in a glass mortar; wash the mixture into a 250 c.c. flask and make up to the mark; the mixture is allowed to stand for 24 hours with intermittent shaking, and it is then filtered; 10 c.c. of the filtrate are evaporated to dryness in a weighed glass dish on the water bath; dried in the water oven and weighed; the weight of the residue obtained multiplied by 500 gives the percentage of the water extract; should this percentage exceed 18 then added sugar is present.

Examples: The dry residue of a sample of cocoa obtained as above was 0·024 gram; then $0{\cdot}024 \times 500 = 12$ per cent. of water extract. The sample does not therefore contain added sugar.

Another sample gave a residue of 0·066 gram; then 0·066 × 500 = 33 per cent. of water extract; the sample contains added sugar.

Estimation of fat. Weigh out 4 grams of the sample into a 100 c.c. flask; add 20 c.c. of strong alcohol and boil the whole on a water bath for three minutes; 20 c.c. of dry ether are now added; the flask is warmed on the water bath, well shaken, cooled and made up to 100 c.c. with dry ether; 2 c.c. of the ether are then added to compensate for the volume of the cocoa, and the contents of the flask are well mixed and allowed to settle; as soon as the contents have settled, 50 c.c. of the clear liquid are put into a flask by a pipette and evaporated to dryness on the water bath and allowed to cool; the residue in the flask is then taken up with ether and passed through a filter paper into a weighed flask; the filter paper is well washed with ether and the filtrate and washings are then evaporated to dryness on the water bath; the residue is dried in the water oven and weighed; the weight of residue obtained multiplied by 50 gives the percentage of fat.

Examples: The weight of fat obtained by the above process was 0·68 gram, then the sample contained 0·68 × 50 = 34·0 per cent. of fat.

Estimation of the added cane sugar. 50 c.c. of the filtrate from the cold water extract are placed in a 100 c.c. flask and 5 c.c. of concentrated hydrochloric acid are added; the flask is placed in water and the temperature is gradually raised until a thermometer in the flask indicates 68° C.; the temperature is maintained between 68 and 70° C. for five minutes; the acid is then neutralized with sodium hydrate solution, and the whole cooled in water and finally made up to 100 c.c.; the copper reducing power is then taken with Fehling's solution, as described under condensed milk (p. 103), and the cane sugar calculated from the weight of cupric oxide obtained by multiplying by 0·4091.

Examples: The weight of copper oxide obtained from 25 c.c. of the inverted solution was 0·130 gram, then 0·130 × 0·4091 × 400 = 21·27 per cent. of cane sugar.

Determination of the ash. 5 grams of the sample are placed

in a platinum dish and carefully ignited over a Bunsen flame until a white residue is obtained; the weight of this residue multiplied by 20 gives the percentage of ash.

Example: The residue obtained from 5 grams of a sample of cocoa was 0·775 gram, then 0·775 × 20 = 1·55 per cent. of ash.

Estimation of the alkalinity of the ash. The ash obtained above is dissolved in water and washed into a beaker; a few drops of methyl orange are added and the contents of the beaker are titrated with N/10 sulphuric acid from a burette until a permanent pink colour is obtained. Each c.c. of N/10 sulphuric acid used = 0·0069 gram of alkalinity calculated as K_2CO_3.

Example: The ash of a cocoa required 16·5 c.c. of N/10 H_2SO_4 to produce a permanent pink colour; then

$$16{\cdot}5 \times 0{\cdot}0069 \times 20 = 2{\cdot}28 \text{ per cent.}$$

of alkalinity calculated as K_2CO_3.

Estimation of the soluble ash. The residue from the cold water solids is gently ignited over a Bunsen flame and the ash weighed; the weight is multiplied by 500 to give the percentage; this should not be less than 2 per cent.

Example: The ash of a sample of cocoa obtained as described weighed 0·005 gram, then 0·005 × 500 = 2·5 per cent.

Estimation of added starch. The presence of added starch in cocoa is easily detected by a microscopical examination; but its exact estimation by chemical methods is almost impossible owing to the presence in cocoa of natural starch in varying quantities; the simplest method for the determination is a microscopical one and the results obtained are as near the true amount as those obtained by the most exact chemical methods. Standard mixtures of cocoa and the starch shown under the microscope are made (5, 10, 15, 20 per cent. etc. of starch) and these are compared under the microscope with slides of the sample; with a little practice the standard containing the same percentage of starch as the sample can easily be picked out.

Estimation of crude fibre. The residue left after the

extraction of fat in the fat determination is washed into a boiling flask of 500 c.c. capacity, with not more than 200 c.c. of water ; 50 c.c. of a 5 per cent. solution of sulphuric acid are added and the whole boiled under a reflux condenser for 30 minutes ; pass the liquid through a filter paper and wash the residue back into the flask with boiling water, not more than 150 c.c. being used ; add 50 c.c. of a 5 per cent. solution of sodium hydroxide and boil under a reflux condenser for 30 minutes ; filter through a dried and weighed filter paper and wash the residue with boiling water, then with very dilute hydrochloric acid solution, followed by very dilute ammonia solution ; the residue is then washed with boiling water until free from ammonia, dried in the water oven and weighed and multiplied by 25 to bring to the percentage.

Crude fibre in a sample of cocoa should not exceed 3·5 per cent. ; any excess over this points to the addition of cocoa husks, which contain about 16 per cent. of crude fibre.

Example : The weight of residue obtained in a crude fibre determination was 0·094 gram, then 0·094 × 25 = 2·45 per cent. of crude fibre.

Typical Analyses of Cocoa (in percentages).

	(1)	(2)	(3)
Water	5·24	11·40	5·68
Fat	33·50	3·80	21·86
Crude fibre	2·73	15·91	1·41
Ash	4·45	7·40	1·55
Soluble ash	2·31	4·55	0·40
Cold water extract ..	12·00	—	33·00
Theobromine	1·00	—	—
Caffeine	0·32	—	—
Cane sugar	—	—	24·98
Starch	10·47	—	32·03

No. (1) is a genuine cocoa of commerce, No. (2) is an analysis of cocoa nibs ; No. (3) is a sample of cocoa adulterated with starch and cane sugar.

CHAPTER V

WHEAT FLOUR, SELF-RAISING FLOUR, BAKING POWDER—BREAD—RICE—STARCHES

WHEAT FLOUR.

Wheat flour is the clean and sound product of ground wheat from which the husks have been removed; it should not contain more than 15 per cent. of water, nor less than 1·5 per cent. of nitrogen, nor more than 1 per cent. of mineral matter. Owing to the demand on the part of the purchasing public for excessive whiteness poor flours are occasionally bleached with oxides of nitrogen. Flours so treated are to be regarded as adulterated under the Sale of Foods and Drugs Acts and are considered to be bleached flours when they contain more than one part per million of nitrite (*Local Government Board Food Reports*, No. 12).

Determination of nitrite (Willard, *Bull. Kansas State Board of Health*, 1906, II, 160). *Solutions required:* A. Weigh out 1 gram of sulphanilic acid and 14·7 grams of glacial acetic acid.

Measure out 286 c.c. of water.

Warm the sulphanilic acid with the acetic acid, diluted with an equal volume of water, until it is dissolved; carefully add the remainder of the water, warming and stirring the mixture to keep the sulphanilic acid in solution.

B. Weigh out 0·2 gram of α-naphthylamine and 14·7 grams of glacial acetic acid.

Measure out 350 c.c. of water.

Warm the naphthylamine with the acetic acid, diluted with twice its volume of water, till it has dissolved, then carefully add the remainder of the water.

The test solution is prepared by mixing equal volumes of A and B.

A cold water extract is made of the sample by digesting the flour with a large quantity of water and filtering; 50 c.c. of the filtrate are placed in a Nessler glass; and in another glass are placed 5 c.c. of a standard potassium nitrite solution (p. 12);

the second glass being filled to the mark with water; 2 c.c. of the reagent are then added to each glass and both the standard and sample are allowed to stand for 15 minutes before comparing the pink colour. The depth of tint is then compared by pouring out either the standard solution or the sample, whichever has the deeper tint, until the columns left in the Nessler glasses are of equal tint.

This test is so sensitive that it cannot always be made in the ordinary laboratory owing to the possible presence of nitrites in the atmosphere which sensibly affect the result.

Total nitrogen. The nitrogen is determined by weighing 0·5 gram and proceeding in the usual way by Kjeldahl's method (p. 85).

Total ash. This is estimated by weighing out 5 grams and burning off all the organic constituents in a furnace; cool and weigh and calculate the result on 100 grams.

Detection of potassium persulphate improver in flour (Hinks, *Analyst*, 1912, XXXVII, p. 91). This is done by first preparing a 3 per cent. solution of benzidine. A portion of the flour is made into a paste with water and the benzidine solution at once poured over; the particles of potassium persulphate become blue coloured.

In connection with the bleaching of flour, the *Reports of the Local Government Board*, 1911 (New Series, No. 49, *Food Reports*, No. 12) by Dr J. M. Hamill "On the bleaching of flour and the addition of so-called improvers to flour," and by Dr G. W. Monier Williams "On the chemical changes produced in flour by bleaching," are of great importance. These reports give references to the important literature of the subject.

SELF-RAISING FLOUR.

Self-raising flour is wheat flour to which the elements of baking powder have been added. About 8 ozs. of sodium bicarbonate and 18 ozs. of cream of tartar to 1 cwt. of flour is the usual proportion; but occasionally phosphate baking powders are used, and, as these sometimes contain more than the allowable amount of calcium sulphate, it is necessary to determine the amount of calcium sulphate in self-raising flour.

Determination of calcium sulphate. Carefully incinerate 5 grams of the sample; dissolve the ash in dilute nitric acid and warm slightly; filter and determine the phosphate with ammonium molybdate as described under vinegar (p. 163). Well mix a further 5 grams of the sample with 1 gram of lime and carefully ignite the mixture; dissolve the residue in dilute hydrochloric acid and add a little bromine water; filter and precipitate the filtrate with barium chloride solution, filter off the precipitated barium sulphate, wash the precipitate well with hot water; dry in the water oven; incinerate and weigh; from the weight obtained subtract the weight of the ash of the filter paper, and deduct the weight of barium sulphate obtained in a blank experiment and multiply the remainder by 0·5837; this gives the calcium sulphate in the sample.

Deduct from the percentage of phosphate found 0·25 per cent. naturally present in flour; multiply the remainder by two which gives pure acid calcium phosphate; add 1/9th, giving an impure phosphate powder containing 10 per cent. calcium sulphate; the calcium sulphate found plus the pure acid calcium phosphate gives the percentage of impure phosphate powder used, and the total calcium sulphate minus the amount constituting the 10 per cent. powder is the excess of calcium sulphate in the self-raising flour.

Example:

Total phosphate found 0·83 per cent.
less 0·25 per cent. natural to flour.
0·58
2
1·16 per cent. pure acid calcium phosphate.
plus 1/9th 0·13
1·29 per cent. of phosphate powder containing 10 per cent. $CaSO_4$.

Pure acid calcium phosphate ..	1·16 per cent.
$CaSO_4$ found	2·32 per cent.
Impure phosphate powder used in flour	3·48 per cent.
Total $CaSO_4$	2·32 per cent.
Due to 10 per cent. phosphate powder	0·13 per cent.
Excess $CaSO_4$	2·19 per cent.

Now 3·48 grams of impure phosphate powder contained 2·32 grams of calcium sulphate, then the original phosphate powder contained 66 per cent. of calcium sulphate

Detection of maize starch. Occasionally the self-raising ingredients are added mixed with maize starch. White (*Analyst*, 1900, xxv, 317) gives the following qualitative test for maize starch ; the sample is thoroughly mixed in a mortar and 1 gram is weighed out into a test-tube with 10 c.c. of water and well shaken ; 15 c.c. of a 1 per cent. solution of sodium hydroxide are then added and the mixture allowed to stand for four hours ; the whole of the wheat starch becomes gelatinized and a drop of the emulsion examined under the microscope shows the granules of maize starch very distinctly.

Estimation of maize starch (Embrey, *Analyst*, 1900, xxv, 310).

Solutions required :

(1) Potassium hydroxide solution containing 18 grams of potassium hydroxide in 1000 c.c.

(2) Iodine solution containing iodine, 0·25 gram ; potassium iodide, 1·00 gram ; water to 250 c.c.

(3) Hydrochloric acid solution containing hydrochloric acid (specific gravity, 1·16), 50 c.c. ; distilled water, 150 c.c.

Weigh out :

(1) 0·2 gram of the sample.

(2) 0·18 gram of pure wheat starch and 0·02 gram of pure maize starch = 10 per cent. maize starch.

(3) 0·17 gram of pure wheat starch and 0·03 gram of pure maize starch = 15 per cent. maize starch.

(4) 0·16 gram of pure wheat starch and 0·04 gram of pure maize starch = 20 per cent. maize starch.

(5) 0·15 gram of pure wheat starch and 0·05 gram of pure maize starch = 25 per cent. maize starch.

(6) 0·14 gram of pure wheat starch and 0·06 gram of pure maize starch = 30 per cent. maize starch.

Place each of the above mixtures in a test-tube, fitted with a cork, add to each mixture 20 c.c. of the potassium hydroxide solution and shake for three minutes ; then add to each 12 drops of the hydrochloric acid solution and centrifuge the mixture ;

remove 1 c.c. of the liquid, avoiding the deposit ; make up to 50 c.c. with water in a Nessler glass and add two drops of the hydrochloric acid solution followed by 1 c.c. of the iodine solution ; thoroughly mix the liquids and compare the tints ; in this way the amount of maize starch can be estimated to within 5 per cent.

BAKING POWDER.

Baking powder is a substance which gives off carbon dioxide on moistening and heating. A number of compounds are used for the purpose all of which are to a greater or less extent mixed with starch. The following are typical compounds used : sodium bicarbonate or ammonium carbonate mixed with cream of tartar, tartaric acid, acid potassium sulphate, acid sodium phosphate, and acid calcium phosphate. The use of acid calcium phosphate and acid potassium sulphate is very objectionable, since the former often contains large quantities of calcium sulphate, and the latter, since with the sodium bicarbonate it forms potassium sulphate and sodium sulphate, both of which have a purgative action. The most satisfactory baking powder is a mixture of sodium bicarbonate or ammonium carbonate with cream of tartar and starch. Until the Sale of Foods and Drugs Act of 1899, baking powder was not subject to analysis as a food, but the definition of food was altered in that Act expressly to include this substance. Adulterations of baking powder are the presence of alum and sulphates (especially calcium sulphate) and excess of starch ; calcium sulphate is generally found in baking powders containing acid calcium phosphate and the presence of this substance in amounts larger than 10 per cent. is to be looked upon as an adulteration (*Local Government Board Food Reports*, No. 13). J. White (*Analyst*, 1902, XXVII, 119) reports the substitution for cream of tartar of a substance having the following composition in percentages.

Moisture ..	4·10	Tartaric acid ..	47·07
Silica ..	0·30	Phosphate ..	6·97
Potash ..	15·50	Sulphate	12·70
Lime ..	9·10	Rice starch ..	5·00

and from which he deduces that the composition was :

Cream of tartar ..	.. 64
Superphosphate ..	.. 31
Rice starch ..	.. 5

The British Pharmacopœia requires that 1 gram of dry cream of tartar should require at least 5·2 c.c. of N/1 sodium hydroxide solution for neutralization, which corresponds to the presence of 97·76 per cent. of cream of tartar ; the above sample required 4·8 c.c. of N/1 sodium hydroxide solution, corresponding to 90·24 per cent. of cream of tartar. Occasionally a substance known as " baking soda " is sold, and it consists of sodium bicarbonate and is very seldom, if ever, adulterated.

Estimation of available carbon dioxide. A Shrotter's carbon dioxide apparatus is carefully cleaned and dried ; concentrated sulphuric acid is poured into the exit tube, in order to dry the carbon dioxide as it escapes ; water is poured into the inlet tube and the whole apparatus is then weighed ; about 1 gram of baking powder is inserted into the bulb and the apparatus is again weighed, the increase in weight is the amount of baking powder taken. The water is now allowed to run into the bulb, when a gentle stream of carbon dioxide is evolved ; after the action has ceased air is sucked through the apparatus, which is then cooled and weighed ; the loss in weight is due to the available carbon dioxide, which has escaped. Dilute hydrochloric acid is now placed in the inlet tube and the apparatus again weighed ; the hydrochloric acid is then run into the bulb and the apparatus warmed on a water bath ; when the reaction has ceased air is again sucked through the apparatus, which is then allowed to cool and weighed ; the loss in weight is due to unavailable carbon dioxide which, with the available carbon dioxide, constitutes the total carbon dioxide present.

Example : In an experiment 0·801 gram of baking powder was used, after the addition of water there was a loss in weight of 0·1065 gram, then the available carbon dioxide

$$= \frac{0{\cdot}0165 \times 100}{0{\cdot}801} = 13{\cdot}33 \text{ per cent.}$$

After the addition of hydrochloric acid there was a further

loss of 0·016 gram, then the unavailable carbon dioxide

$$= \frac{0{\cdot}016 \times 100}{0{\cdot}801} = 1{\cdot}99 \text{ per cent.}$$

and the total carbon dioxide = 1·99 + 13·33 = 15·32 per cent. Baking powder should contain at least 8 per cent. of available carbon dioxide.

Estimation of sulphates. 5 grams of the sample are dissolved in dilute hydrochloric acid by continuous boiling; a slight excess of barium chloride solution is added and the precipitate of barium sulphate allowed to settle; the solution is then carefully filtered, the precipitate is well washed, dried in a water oven, incinerated and weighed; the weight of ash, less the weight of the ash of the filter paper, multiplied by 0·3434 gives the weight of sulphates (as SO_3) in 5 grams of the sample.

Estimation of calcium. 5 grams of the sample are dissolved, with boiling, in dilute hydrochloric acid; a slight excess of ammonia is added, followed by a slight excess of acetic acid; any precipitate is filtered off, and to the filtrate is added an excess of ammonium oxalate; the precipitate of calcium oxalate is collected on a filter paper, washed, dried in a water oven, incinerated, ignited over a blowpipe, cooled, and weighed; the weight of the ash is the weight of calcium (as CaO) in 5 grams of the sample.

Estimation of tartaric acid. White's modification (*Analyst*, 1902, XXVII, 119) of Goldenberg's method (*Analyst*, 1896, XXI, 333). 2 grams of the sample are dissolved in 20 c.c. of hot water and 5 c.c. of dilute hydrochloric acid and made up to 100 c.c.; of this 50 c.c. are made faintly alkaline with solid potassium carbonate, boiled for two minutes, cooled and made up to 100 c.c. Place 50 c.c. (= 0·5 gram of the powder) in a 500 c.c. flask with 10 c.c. of glacial acetic acid and 250 c.c. of a mixture of equal parts of alcohol (90 per cent.) and ether (sp. gr. ·730); close the flask with a well fitting cork; shake the contents well and allow them to stand for three hours; filter, wash the precipitate with the alcohol and ether mixture until the washings are neutral to litmus; dissolve the

precipitate off the filter paper with boiling water and finally titrate with N/10 sodium hydroxide solution; each c.c. of N/10 sodium hydroxide solution used represents 0·0075 gram of tartaric acid or 0·0188 gram of cream of tartar.

Estimation of lead in tartaric acid and cream of tartar, and citric acid (Tatlock and Thomson, *Analyst*, 1908, XXXIII, 173). 10 grams of cream of tartar are treated with 50 c.c. of water and 40 c.c. of 2N ammonia (in the case of tartaric acid 81 c.c. of 2N ammonia and 9 c.c. of water, and in the case of citric acid 85 c.c. of 2N ammonia and 5 c.c. of water are used); agitate the mixture well until solution is complete; filter through a dry filter paper; to 50 c.c. of the filtrate add 0·1 gram of potassium cyanide and 1 c.c. of a strong colourless solution of ammonium sulphide, and compare with the colours obtained from a standard lead solution (see p. 16).

The *Report to the Local Government Board*, 1911 (New Series, No. 46, *Food Reports*, No. 13) by Dr J. M. Hamill "On the presence of calcium sulphate in baking powder and self-raising flour" should also be consulted.

BREAD.

Bread is the substance obtained by moistening, kneading and baking wheat flour, and to which porosity has been imparted by means of carbonic acid gas, obtained from baking powder, or by means of fermentation produced in the dough by the addition of yeast. Bread should not contain more than 45 per cent. of moisture, nor more than 1·5 per cent. of ash, nor more than 0·2 per cent. of ash insoluble in hydrochloric acid. In estimating the moisture in bread care must be taken to obtain a representative sample, as bread contains less moisture on the outside than in the middle of the loaf. The addition of foreign starches, in the form of boiled potatoes, has been practised, but this adulteration is difficult if not impossible to detect owing to the alteration in the starch granules produced by the baking process. Occasionally still alum is added to bread to produce that whiteness which the purchasing public considers to be synonymous with purity.

Detection of alum. *Solutions required :*

(1) Logwood solution. A freshly prepared alcoholic extract of logwood chips.

(2) A saturated solution of ammonium carbonate.

Dilute 5 c.c. of the logwood extract and 5 c.c. of the ammonium carbonate solution to 100 c.c., and pour the mixture over about 10 grams of bread crumbs in a small porcelain dish ; allow the mixture to stand for about five minutes ; pour off the excess of liquid and dry the residue in the water oven ; in the presence of alum, a lavender blue colour is produced, and in its absence, a reddish brown colour is obtained. Care must be taken that the bread has not become sour before the test is performed, as sour bread will occasionally give the alum colour. Care must also be taken that the logwood extract is always freshly made.

Estimation of alum. Carefully incinerate 100 grams of bread crumbs over a Bunsen flame, completing the process by the addition of a small quantity of sodium carbonate and potassium nitrate ; dissolve the residue in water and acidify with dilute hydrochloric acid and evaporate to dryness in a platinum dish ; moisten the residue with hydrochloric acid and dissolve in water ; filter and wash the precipitate well with hot water ; dry in the water oven, ignite, weigh and record the weight as silica (SiO_2).

The filtrate from the silica determination is poured into an excess of concentrated sodium hydroxide solution, boiled and filtered ; acidify the filtrate with hydrochloric acid ; add a few drops of sodium phosphate solution, followed by excess of ammonia, filter, well wash the precipitate, dry in a water oven, ignite and weigh ; the weight obtained multiplied by 1·0397 gives the grains of ammonium alum per four pound loaf ; allowance must be made for the aluminium phosphate naturally present in the bread, amounting to about 15 to 18 grains per four pound loaf.

The *Report of the Local Government Board* (New Series, No. 55) by Dr Hamill " On the nutritive value of bread made from different wheat flours " is of considerable value as regards the subject of nutrition.

RICE.

Rice is the hulled grain of *Oriza sativa*; the lower grades are sometimes polished to give them an appearance of higher quality and this is produced by the application of talc to the grains; the talc is generally mixed with glucose, glycerine and starch paste, and occasionally with mineral oil. Talc is a silicate of magnesium with small quantities of the silicates of iron, aluminium and potassium, and its general use for this purpose is not advisable; should the ash of a sample of rice exceed 0·3 per cent. it is advisable to look for a coating of talc. The limit of steatite or talc suggested by the Local Government Board is 0·5 per cent.

Estimation of talc. A number of methods have been devised for the estimation of talc, but the simplest is that recommended by E. W. T. Jones (*Chem. News*, 1913, CVIII, 176) in which 5 grams of rice are placed in a squat 150 c.c. beaker, 20 c.c. of ether are added and the whole agitated for a few minutes; the ether is poured into a 100 c.c. beaker, containing about 2 c.c. of water, and then evaporated off on a water bath; the remaining ether is also evaporated from the rice grains and about 15 c.c. of cold distilled water added; the whole is agitated and the water added to the ether residue and repeated four or five times until the water comes away clear; the mixed washings are allowed to stand overnight; the clear supernatant liquid is poured off, leaving the talc and a little fine rice behind, which is transferred to a weighed platinum dish with methylated spirit, evaporated to dryness on a water bath, ignited and weighed; the weight obtained is the weight of talc in 5 grams of the sample.

The following analyses of various samples of rice have been supplied by Mr West Knights. The numbers represent percentages.

	(1)	(2)	(3)	(4)	(5)	(6)
Ash	0·44	0·54	0·42	0·54	0·90	1·20
Steatite washed from the surface of the rice	0·14	0·16	0·09	0·23	0·47	0·75

All the samples except (6) are within the limit; No. (6) is of questionable genuineness.

STARCHES.

The general appearance of the various starches under the microscope is an essential part of the examination of foods. A small portion of the starch should be placed in a small glass mortar and well mixed with water; a drop of the mixture is then placed on a glass slide, covered with a slip and examined with a 2/3 and 1/6 lens or a microscope. After a little practice the characteristic appearance of the various commoner starches becomes familiar and mixtures of starches can readily be distinguished.

Microscopical Appearance of the Commoner Starches.

1. *Potato.* This starch consists of oval granules, having an annular hilum at the smaller end and very distinct and complete rings; some of the granules are very large and some are very small.

2. *Arrowroot.* Not unlike potato, but the hilum is usually at the larger end.

3. *Wheat.* The granules are circular and flat; they seldom exhibit a hilum and are very variable in size; some are very large and some very small with a few intermediate in size.

4. *Maize.* The granules are polygonal and exhibit facets and a star-shaped hilum; under polarized light they show a well marked and characteristic cross.

5. *Barley.* This starch has circular granules, very similar to those of wheat, but slightly smaller; they exhibit no hilum, but some have rings.

6. *Rye.* The granules are not unlike barley, and some of them show a broken hilum.

7. *Rice.* This consists of very small polygonal granules, with some compound ones.

8. *Oat.* This starch also consists of polygonal granules, slightly larger than those of rice; there are usually a number of compound granules and under a very high power they exhibit a hilum.

9. *Sago.* The granules of this starch are oval and they exhibit a circular hilum and have faint rings.

10. *Tapioca*. The granules are "top hat" shaped, truncated at one end; they have a well marked hilum.

11. *Pea* or *Bean*. The large pea granules have a broken longitudinal hilum in the centre and the bean granules are similar, except that perhaps they are a little larger and more uniform.

CHAPTER VI

PEPPER, CAYENNE, MUSTARD—NUTMEG, CINNAMON, GINGER

PEPPER.

Pepper is the dried berry of *Piper nigrum*; black pepper is the immature whole berry, sun-dried or roasted and ground; white pepper is the mature berry from which the outer coating has been removed.

The chief adulterants of pepper are: excess of mineral matter (sand, etc.), starch (chiefly rice), long pepper, added husk, and ground olive stones or pepperette or poivrette. It must be stated that the public themselves are primarily responsible for the adulteration of pepper, since of late years a craze has grown for excessive whiteness in pepper.

Detection of adulteration. The addition of starch is detected under the microscope and the amount of foreign starch determined by comparison with standard slides as described under cocoa (p. 127); ground olive stones and long pepper may also be detected by a microscopical examination, olive stones showing bright with a red tint under polarized light, and long pepper a bluish tint. In a routine analysis the determinations usually made are the total ash, the ash insoluble in hydrochloric acid and the crude fibre; the total ash of a black pepper should not exceed 5·5 per cent. and of a white pepper 3 per cent.; the crude fibre should not exceed 5 per cent. Campbell Brown (*Analyst*, 1887, XIII, 24) has shown that the crude fibre of poivrette is 48·48 per cent., of ground almond shells 51·68 per cent. and of olive stones 45·38 per cent.

Detection of poivrette (Martelli, *Staz. Sper. Ag. Ital.* XXVIII, 53). Digest for two or three days 1 gram of phloro-glucinol in 50 to 60 c.c. of hydrochloric acid (specific gravity 1·1) and decant the clear solution. To about 0·5 gram of the sample of pepper add sufficient of the reagent to cover it and heat cautiously until fumes of hydrochloric acid begin to come off; poivrette and similar substances (*e.g.* almond shells, walnuts, etc.) give a very intense cherry red colour easily distinguished by the naked eye from the yellow colour of pepper. On adding to the mass a little water and decanting the liquid a violet red powder is left, which consists almost entirely of poivrette stained by the reagent.

Detection of turmeric (Parry, *Chem. and Drugg.* 1914, LXXXIV, p. 34). *Solution required:*

Weigh 1 gram diphenylamine.

Measure 20 c.c. of 90 per cent. alcohol,
25 c.c. of sulphuric acid.

Dissolve the diphenylamine in the alcohol, add the sulphuric acid and cool. One drop of the reagent is placed on a microscopical slide, and a small quantity of the sample on a cover glass; the cover glass is then mounted over the slide so that the sample falls on the reagent; the particles of turmeric will be found to be coloured a fine purple. The proportion of turmeric present in the pepper is estimated by comparison with slides containing a known amount.

Typical Analyses of Pepper (in percentages).

	A	B	C
Water	9·83	10·60	9·71
Ash	3·70	1·34	1·35
Volatile oil	1·60	1·26	1·47
Starch	37·30	43·10	31·45
Crude fibre	10·02	4·20	3·92

A is a sample of Singapore black pepper, B of Singapore white pepper, C of commercial white pepper.

CAYENNE.

Cayenne is the dried and powdered or ground fruit of *Capsicum fastigiatum*, *Capsicum frutescens*, or of *Capsicum annum*; it is very rarely adulterated in England, but occasionally foreign starch, mineral matter and ground olive stones have been found. The presence of foreign starch is detected by a microscopical examination; excessive mineral matter by the ash, and the ash insoluble in hydrochloric acid; the ash of a genuine sample of cayenne should not exceed 7 per cent. and the ash insoluble in hydrochloric acid 0·5 per cent.; a determination of the crude fibre will show the presence of ground olive stones, genuine cayenne having a crude fibre of not more than 28 per cent.

A typical Analysis (in percentages) of a genuine Cayenne by Richardson (U.S. Dept. of Agric. Bull. XII, p. 211).

Water	5·74
Ash	5·24
Volatile oil	17·90
Fixed oil	1·58
Crude fibre	18·10
Albuminoids	11·20

MUSTARD.

Mustard is the ground seed of *Sinapis alba*, *Brassica juncea* or *Brassica nigra*; after grinding the mass is sifted and a large part of the oil removed; the pungent properties of mustard are due to the presence of allyl isothiocyanate, which is produced by the action of the enzyme myrosin and water on the potassium myronate present in the seed. The most common adulterants of mustard are starch and turmeric; in the natural state mustard should contain no starch, but a practice has arisen of mixing mustard with comparatively large quantities of starch, the manufacturer stating that the presence of starch is necessary to prevent decomposition and caking and that the customers prefer it so mixed; but this contention has no foundation in fact, the mixture is usually sold as "Mustard Condiment."

In the routine examination of mustard, the presence of starch is detected by the microscope and the percentage by comparison with standard slides.

Detection of turmeric. A small portion of the sample is treated with ammonia and examined under a magnifying glass, in the presence of turmeric some of the grains will be coloured red ; or a strip of filter paper may be soaked in an alcoholic extract of the sample, then in a solution of borax, finally acidified with hydrochloric acid and allowed to dry ; in the presence of turmeric a rose red colour is produced, which becomes a deep blue when treated with ammonia or a green when treated with dilute potassium hydroxide solution.

Estimation of oil. 5 grams of the mustard are placed in a Soxhlet thimble in a Soxhlet extractor and treated with ether on a water bath until the ether passes over colourless, when the flask is removed and the ether evaporated off and the residue dried in a water oven to constant weight.

In a sample from which the oil has not been removed, the percentage of mustard may be calculated roughly from the formula

$$\frac{\text{per cent. of oil} \times 100}{30}.$$

Typical Analyses of Mustard (in percentages).

	A	B	C
Moisture	5·55	6·83	6·43
Total ash	5·58	5·03	4·37
Ether extract	17·46	13·81	27·45
Alcohol extract	25·31	14·21	16·31
Volatile oil	3·98	2·26	0·00
Crude fibre	3·28	10·90	4·95

A was a sample of commercial English brown mustard flour, freed from husk, and with a portion of the fixed oil removed ; B was a sample of English brown mustard husk ; C was a sample of whole ground mustard seed.

NUTMEG.

Nutmeg is the fruit of *Myristica fragrans*, artificially dried or sun-dried and deprived of its testa ; when the fruit contains its testa it is known as mace ; owing to the fact that nutmeg

is generally sold as a whole fruit, adulteration is practically impossible and only becomes possible when the sample is ground. In the routine examination of nutmeg the usual determinations are the ash, which should not exceed 5 per cent.; the ash insoluble in hydrochloric acid, which should not exceed 0·5 per cent.; and the crude fibre, which should not exceed 10 per cent.

CINNAMON.

Cinnamon is the bark of *Cinnamomum zelanicum*, from which the outer layers have been removed, for which cassia or the dried bark of *Cinnamomum cassia* is occasionally substituted; this substitution is easily detected by the naked eye, cinnamon being thin and containing no outer bark, whereas cassia is thick and contains the outer bark. The larger portion of cinnamon is obtained from Ceylon, cassia being grown in China or India; cinnamon is chiefly required for the volatile oil which it contains. Dyer and Gilbard (*Analyst*, 1895, xx, 129) have found cinnamon adulterated with ground walnut shells. In the routine examination of cinnamon, the ash, which should not exceed 6 per cent., is determined and the sample is examined microscopically.

Typical Analyses (*in percentages*).

	A	B
Moisture	12·41	9·97
Total ash	4·22	0·87
Ash soluble in water ..	0·46	0·37
Ash insoluble in water ..	2·76	0·50
Fibre	34·25	47·67

These analyses are by Dyer and Gilbard (*Analyst*, 1895, xx, 129); A is of cinnamon and B of ground walnut shells.

GINGER.

Ginger is the dried rhizome of *Zingiber officinale*, from India, China or Jamaica, washed and sun-dried, and sometimes scraped and immersed in a solution of lime which prevents the destruction of the rhizome from the attacks of insects. The

most common adulterants of ginger are exhausted ginger, excessive mineral matter, foreign starches, turmeric and capsicum. The usual determinations are crude fibre (not more than 8 per cent.), total ash (not more than 8 per cent.), lime (not more than 1 per cent.), ash insoluble in hydrochloric acid (not more than 3 per cent.) and the aqueous extract.

Detection of capsicum (Garnett and Crier, *Pharm. Jour.* 1909, vol. LXXXVII, p. 441). A small portion of the sample is heated with a dilute solution of potassium hydroxide which destroys the pungent odour of ginger but not of capsicum.

Determination of the ash. 5 grams of the sample are incinerated in a platinum dish over a Bunsen burner and weighed; the weight of ash multiplied by 20 gives the percentage of total ash; the ash is then extracted with hot water, the solution is filtered, the residue on the filter paper is well washed with hot water, dried in a water oven and incinerated; the weight of the ash, less the weight of the ash of the filter paper, multiplied by 20 gives the percentage of insoluble ash, and this deducted from the total ash gives the percentage of soluble ash.

Determination of the cold water extract (Winton, Ogden and Mitchell, *U.S. Dept. of Agric. Bull.* LXV, p. 59). Four grams of the sample are placed in a 200 c.c. graduated flask and water is added to the mark; the mixture is shaken at intervals for 8 hours and then allowed to stand for 16 hours. The contents of the flask are then filtered and 50 c.c. of the filtrate are evaporated to dryness, dried in the water oven and weighed; the weight of the residue multiplied by 100 gives the cold water extract.

Detection of exhausted ginger. Commercially there are two varieties of exhausted ginger, one the product left after the alcoholic extraction for the preparation of ginger extract, and the other the product of the water extraction in the preparation of ginger beer, etc. Dyer and Gilbard (*Analyst*, 1893, XVIII, p. 197) have shown that the ash soluble in water is a reliable method of detecting exhausted ginger, since the water soluble ash of a genuine ginger varies from 1·9 to 3 per cent., whereas an exhausted ginger gives a water soluble ash of from 0·2 to 0·5 per cent.

Detection of foreign starches, etc. The microscopic appearance of ginger is so characteristic that adulteration with foreign starches has fallen into disuse; but a microscopical examination should always be made for foreign substances.

Typical Analyses (in percentages).

	(1)	(2)
Moisture	13·6	12·1
Volatile oil	0·7	0·8
Fixed oil	3·0	5·2
Alcoholic extract ..	3·1	1·2
Total ash	3·1	2·1
Soluble ash	2·4	0·4
Insoluble ash	0·7	1·7

No. (1) is a genuine ginger and No. (2) is an exhausted ginger.

CHAPTER VII

CANE SUGAR—GOLDEN SYRUP AND TREACLE—HONEY—JAM

CANE SUGAR.

Cane sugar is found in the sap and fruits of a very large number of plants. Its chief manufacturing sources are the beet (*Beta vulgaris*), the sugar cane (*Saccharum officinarum*) and the sugar maple (*Acer saccharinum*). When heated to 160° C. cane sugar forms a light amber coloured mixture of dextrose and lævulose, known as "barley sugar" and when heated above this temperature it forms caramel. Adulteration of cane sugar is very rare; occasionally ultramarine blue is added to correct the yellow colour, and coloured crystals are sold in substitution for Demerara sugar, which is or should be manufactured in the island of Demerara in the West Indies. The colour is imparted to Demerara sugar by the addition of

tin chloride, which fixes the natural colour on the sugar crystals and is itself almost completely removed in the molasses. In the manufacture of sugar, the beet or sugar cane is first cleansed from mud and stones and then sliced ; and the juice extracted by pressure or diffusion. The juice is then clarified by the addition of lime to the warm solution ; the excess of lime is precipitated by means of CO_2 ; the juice, which has now an amber colour, is concentrated with exhaust steam and finally crystallized in " vacuum pans " ; the crystals are separated from the molasses by centrifugal force and the molasses then treated with strontium hydroxide, which forms with the saccharose a precipitate of insoluble strontium saccharate ; this is decomposed with CO_2 giving a further crop of cane sugar crystals.

Detection of ultramarine blue. 50 grams of the sample of sugar are dissolved in water and allowed to stand overnight, when the colouring matter will sink to the bottom and should be washed by decantation ; the residue is then treated with hydrochloric acid, when the blue colour will be discharged with evolution of sulphuretted hydrogen.

Detection of dyed crystals. The sample is extracted with alcohol (90 per cent.) ; the solution is filtered and evaporated to dryness and the residue taken up with alcohol ; a skein of wool is then placed in the solution and the whole placed on a water bath for 30 minutes ; the wool is then removed and dried ; in the presence of artificial dye the wool will be coloured yellow.

GOLDEN SYRUP AND TREACLE.

Golden syrup and treacle are bye products in the manufacture of cane sugar and consist principally of cane sugar, dextrose and lævulose. They contain from 30 to 35 per cent. of cane sugar, which cannot be separated by direct crystallization and they should not contain more than 20 per cent. of water. They are frequently adulterated by the addition of starch-glucose, which is the product obtained by the hydrolysing of starch until the greater part has been converted into dextrose.

Analysis of golden syrup and treacle. The most convenient method of analysis for these products is that devised by E. W. T. Jones (*Analyst*, 1900, xxv, 67).

Estimation of total solid matter. 25 grams of the sample are dissolved in water and made up to 250 c.c.; the specific gravity of this solution (which should not be less than 1032, indicating 20 per cent. of water) is taken with the specific gravity bottle, and the excess gravity over 1000 is divided by four, giving the total solid matter. Jones finds that this gives a more accurate practical figure than that obtained by dividing by 3·86 which is theoretically correct. The percentage of total solid matter subtracted from 100 gives the water.

Example: The specific gravity of a 10 per cent. solution of a sample of golden syrup was 1032·7, then the sample contained $\frac{32{\cdot}7}{4} = 8{\cdot}175 \times 10 = 81{\cdot}75$ per cent. of solid matter, and $100 - 81{\cdot}75 = 18{\cdot}25$ per cent. of water.

The amount of water in the sample cannot be determined in the usual way by drying a weighed quantity of the sample in the water oven, owing to the impossibility of removing all the water.

Estimation of cane sugar and starch glucose. The temperature of the 10 per cent. solution is raised to 17·5° C.; the solution is examined in the polarimeter tube (100 mm.) and the angle of rotation noted; 50 c.c. of the 10 per cent. solution are then heated with 5 c.c. of N/1 hydrochloric acid in a 100 c.c. flask in a boiling water bath for 20 minutes; the solution is then cooled; 5 c.c. of N/1 sodium hydroxide solution are added and the whole is made up to 100 c.c. at 15·5° C.; the resulting solution is then raised to 17·5° C. and the reading taken in the polarimeter and multiplied by two to correspond to the 10 per cent. solution. If the sample after inversion has a dextro-rotation, starch glucose is present and its amount is calculated from the formula

$$\frac{([\alpha]_D \text{ after inversion} + 11) \times 100}{120}.$$

The amount of cane sugar is calculated from the alteration

in the specific rotatory power after inversion. The specific rotatory power of cane sugar is $[\alpha]_D + 66{\cdot}5°$, and that of invert sugar is $[\alpha]_D - 19{\cdot}6°$; then the amount of cane sugar in a sample is calculated from the formula

$$\frac{100 \times ([\alpha]_D \text{ after inversion} - [\alpha]_D \text{ before inversion})}{[\alpha]_D \text{ of cane sugar} - [\alpha]_D \text{ invert sugar}}.$$

Example: A sample of syrup gave a specific rotatory power after inversion of $[\alpha]_D = +11{\cdot}07$

then $$\frac{(11{\cdot}07 + 11) \times 100}{120} = 18{\cdot}4 \text{ per cent.}$$

of starch glucose in the sample. The sample was said by the manufacturers to contain 18 per cent. of glucose syrup.

A sample of syrup gave $[\alpha]_D$ before inversion + 19·0, and after inversion $[\alpha]_D - 12{\cdot}5$; then the cane sugar in the sample was

$$\frac{100\,(19{\cdot}0 - 12{\cdot}5)}{66{\cdot}5 - 19{\cdot}6} = 36{\cdot}62 \text{ per cent.}$$

Determination of the copper reducing power. 10 c.c. of the 10 per cent. solution are made up to 100 c.c., and 20 c.c. of this solution are added to 50 c.c. of Fehling solution, diluted with 30 c.c. of water, in a boiling water bath; the mixed solutions are allowed to remain in the water bath for 12 minutes and then filtered; the precipitate is well washed, dried in a water oven, ignited in a porcelain crucible and weighed as CuO; the weight of CuO obtained multiplied by 0·4307 gives the invert sugar.

Example: The weight of CuO obtained in an experiment was 0·210, then $0{\cdot}210 \times 0{\cdot}4307 = 0{\cdot}090447 \times 500 = 45{\cdot}22$ per cent. of invert sugar.

10 c.c. of the 10 per cent. solution are now inverted as described above and made up to 100 c.c.; 20 c.c. of this solution are added to 50 c.c. of Fehling solution as before. The weight of CuO obtained in the first experiment is deducted from that obtained in the second experiment, and the difference multiplied by 0·4091 gives the cane sugar.

Example: After inversion the sample of syrup gave 0·366 gram of CuO, then $0{\cdot}366 - 0{\cdot}210 = 0{\cdot}156 \times 0{\cdot}4091 = 0{\cdot}0638196 \times 500 = 31{\cdot}91$ per cent. of cane sugar.

HONEY.

Honey is the saccharine exudation of plants which has been collected, modified and stored in the honey comb by the honey bee (*Apis mellifica*). It consists principally of dextrose and lævulose in roughly equal proportions and a small quantity of cane sugar. It should not contain more than 25 per cent. of water. Honey is extensively adulterated chiefly with cane sugar or starch glucose. A 10 per cent. solution of honey examined in the polarimeter should always show a lævo-rotation. In the event of a sample showing a dextro-rotation, either it contains added cane sugar, or it is artificial honey, or it contains starch glucose. The analysis of honey is carried out exactly as if it were a sample of golden syrup. A solution of the sample should be tested for calcium with ammonia and ammonium oxalate, and for sulphates with hydrochloric acid and barium chloride, as the presence of calcium sulphate is an indication of the presence of starch glucose.

Some typical analyses of various sugars (in percentages).

	(1)	(2)	(3)	(4)	(5)	(6)
Cane sugar	32·71	22·39	1·78	36·49	85·92	2·29
Invert sugar	44·91	20·45	77·12	32·14	6·84	71·43
Starch glucose	—	35·35	—	—	—	—
Water	18·29	20·06	20·75	20·32	5·37	21·64
Ash	3·00	1·40	0·22	8·45	1·37	4·15
Total solid matter ..	81·71	79·94	79·27	79·56	94·63	78·36

No. (1) is a sample of golden syrup; No. (2) is a sample of golden syrup containing 35 per cent. of starch glucose; No. (3) is a sample of honey; No. (4) is a sample of treacle; No. (5) is a commercial cane sugar; and No. (6) is an artificial invert sugar.

JAM.

Jam is a mixture prepared by boiling fruit pulp. Jams of the highest quality contain nothing but the pulp and juice of the fruit after which they are named and pure cane sugar. The pulp mixed with the necessary proportion of water is boiled and varying amounts of sugar are added, from one-third to one-half of the whole; the final product is solid, caused by the gelatinization which has taken place owing to the presence of pectin in the juice.

Adulterations. In the case of some fruits, notably raspberry and strawberry, there is present only a small quantity of pectin, and it is therefore necessary, in order to obtain the correct consistency in the finished product, either to boil the mixture for a considerable time, which spoils the appearance, or to add some fruit juice rich in pectin, as for example apple or gooseberry juice. Provided that this addition is notified on the label, it cannot be regarded as an adulteration, since it is not added for the purpose of increasing the bulk. At the same time it must be noticed that some manufacturers take advantage of this fact to add comparatively large quantities of apple pulp, and this must be regarded as an adulteration. Occasionally also, for the sake of cheapness, commercial glucose is used instead of cane sugar.

Estimation of water. The determination of water in jam is exceedingly difficult, owing to the practical impossibility of driving off the last traces of water. The sugars, estimated by Fehling's process, plus 6 per cent. allowed for fibre and other constituents, deducted from 100, give a fair approximation of the amount present. A jam should not contain more than 50 per cent. of water.

Estimation of glucose. 10 grams of the jam are dissolved in about 70 c.c. of water in a 200 c.c. flask; 7 c.c. of concentrated hydrochloric acid are added; the mixture is heated to 68° C. and kept at that temperature for five minutes; the solution is then cooled and neutralized with sodium hydroxide solution; 10 c.c. of lead subacetate solution are added to clarify the mixture which is then made up to 200 c.c., well shaken and filtered; the filtrate is placed in a polarimeter tube and the angle of rotation observed at 87° C. at which temperature the reading of invert sugar is 0, all rotation therefore is due to glucose. The average specific rotatory power $[\alpha]_D$ of glucose is 150, and from this the percentage of glucose is calculated.

Example: A sample of jam had a specific rotatory power $[\alpha]_D$ of 30° at 87° C., then the glucose in the sample

$$= \frac{30 \times 100}{150} = 20 \text{ per cent.}$$

Detection of apple pulp. About 5 grams of the jam are boiled with 25 c.c. of water and then cooled ; a few drops of iodine solution are added, when, in the presence of apple pulp, some of the cells appear blue, owing to the presence of starch in the cells of apple pulp. The microscopic appearance of apple pulp should be carefully studied, and the percentage estimated by comparison, under the microscope, with jams containing a known amount of the pulp.

Estimation of salicylic acid. Harry and Mummery (*Analyst*, 1905, xxx, 124) have devised a convenient method of estimating salicylic acid in jam. 50 grams of the sample are placed in a 300 c.c. flask ; a small quantity of water is added, followed by 15 to 20 c.c. of a saturated solution of basic acetate of lead, and the whole is then made alkaline with 15 to 20 c.c. of N/1 sodium hydroxide solution ; the alkali throws down excess of lead, and afterwards dissolves the hydroxide and some albuminous material ; and, in order to reprecipitate these, 15 to 20 c.c. of N/1 hydrochloric acid are added ; the contents of the flask are now made up to 300 c.c., well shaken and filtered ; 200 c.c. of the filtrate are acidified with hydrochloric acid, filtered if necessary, and extracted three times with ether ; the mixed ether extracts are evaporated and the salicylic acid is dissolved in a small quantity of dilute alcohol and made up to 100 c.c. ; 50 c.c. of this solution are placed in a Nessler glass, 2 c.c. of neutral ferric chloride solution are added and the amount of salicylic acid present estimated by comparison with the colour obtained from known quantities of a standard solution of salicylic acid.

Example : 50 c.c. of the alcoholic solution gave a colour exactly matched by 1·5 c.c. of standard salicylic acid solution (containing 0·1 per cent. of salicylic acid) ; then the amount of salicylic acid in the sample is

$$\frac{1{\cdot}5 \times 0{\cdot}001 \times 2 \times 3 \times 2}{2} = 0{\cdot}009 \text{ per cent.}$$

or 0·63 grain per pound.

Table containing the specific rotatory powers of various sugars.

	$[\alpha]_D$
Cane sugar ($C_{12}H_{22}O_{11}$)	+ 66·6
Maltose ($C_{12}H_{22}O_{11}$)	+138
Lactose Anhydrous ($C_{12}H_{22}O_{11}$)	+ 55·4
Lactose (cryst. $C_{12}H_{22}O_{11}H_2O$) ..	+ 52·6
Dextrose ($C_6H_{12}O_6$)	+ 51·3
Lævulose ($C_6H_{12}O_6$)	− 95·4
Invert sugar ($C_6H_{12}O_6)_2$.. ..	− 24·4

Table containing the weights of various sugars equivalent to 50 *c.c. of Fehling's solution, and to* 1 *gram of CuO.*

50 c.c. of Fehling's solution			= 0·2375	gram	of dextrose.
50	,,	,,	= 0·2572	,,	lævulose.
50	,,	,,	= 0·2470	,,	invert sugar.
50	,,	,,	= 0·3380	,,	lactose.
50	,,	,,	= 0·3890	,,	maltose.

1	gram	of CuO	= 0·7435	gram	maltose.
1	,,	,,	= 0·4535	,,	dextrose.
1	,,	,,	= 0·4715	,,	invert sugar.
1	,,	,,	= 0·4897	,,	lactose.
1	,,	,,	= 0·4911	,,	lævulose.

CHAPTER VIII

ALCOHOLIC BEVERAGES—WHISKEY, RUM, GIN, BRANDY —BEER—CIDER

SPIRITS.

Whiskey. Whiskey is principally manufactured in Scotland from malted and unmalted barley, and from malted and unmalted barley and other grains in Ireland. The flavour and bouquet of the spirit are due to the presence of "impurities," higher alcohols, esters, etc. The spirit is produced by the distillation of a fermented extract of the grain. In Ireland the spirit as distilled has an alcoholic strength of about 30 degrees over proof, and in Scotland about 25 degrees over proof. The distillation is carried out either in a "pot" or a "patent" still, and the character of the distillate is dependent on the still used. "Patent" still spirit is comparatively free from the higher alcohols, esters, etc., and it was considered by analysts to be adulterated until the Royal Commission on Whiskey

and other Potable Spirits, 1908–9, reported to the contrary. The crude distillate is allowed to mature in wooden casks for a number of years. The Sale of Foods and Drugs Act, 1879, requires that whiskey shall not be sold at a greater dilution than 25 degrees under proof, and, practically, the only adulteration now is an excessive dilution, since the Royal Commission on Whiskey and other Potable Spirits, 1908–9, reported that "Whiskey is applicable to a spirit manufactured from malt or from malted and unmalted barley or other cereals" and that "Whiskey is a spirit obtained by distillation from a mash of cereal grains saccharified by the diastase of malt." The amount of alcohol in whiskey may vary between 50 and 55 per cent.

Proof spirit is that spirit which when mixed with gunpowder will just allow the gunpowder to be ignited; if the gunpowder burns with a flash the spirit is over proof, and if the gunpowder cannot be ignited the spirit is under proof.

Rum. Rum is produced in the West Indies, principally Jamaica, from molasses by fermentation and distillation. The "impurities" in rum are usually comparatively high; the colouring matter consists of caramel, and the average alcoholic strength of imported rum is 25 degrees over proof. The Sale of Foods and Drugs Act, 1879, requires that rum shall not be sold at a greater dilution than 25 degrees under proof, and, practically, the only adulteration is excessive dilution. The amount of alcohol in rum varies between 50 and 60 per cent.

Gin. Gin is manufactured by the distillation of spirit, obtained by the fermentation of grain extract, with Juniper berries. The chief grains used are maize malt and rye. Sweetened gin is obtained by adding syrup to plain gin. The Sale of Foods and Drugs Act, 1879, requires that gin shall not be sold at a greater dilution than 35 degrees under proof, and adulteration other than excessive dilution is very uncommon. The amount of alcohol in gin is usually between 45 and 55 per cent.

Brandy. Brandy is, or should be, made from the juice of the grape. It contains higher alcohols, esters, volatile oil, tannin and colouring matter. The colour is obtained by storing

the colourless fresh spirit in oak casks. When it is first distilled brandy has an alcoholic strength of about 25 degrees over proof; the standard alcoholic strength for brandy in the United States of America is 19 degrees over proof. The Sale of Foods and Drugs Act, 1879, requires that brandy shall not be sold at a greater dilution than 25 degrees under proof. The most common adulterations are excessive dilution and blending with "silent" spirit. British brandy is a spirit obtained from grains with flavouring ingredients added. The amount of alcohol in brandy is between 45 and 55 per cent.

Estimation of the alcoholic strength of spirits. The specific gravity of the sample at 60° F. is taken with a specific gravity bottle; 100 c.c. of the sample are placed in a distilling flask of not more than 250 c.c. capacity; 25 c.c. of water are added and the contents of the flask distilled until about 95 c.c. have passed over; the distillate is collected in a 100 c.c. flask, cooled to 60° F. and made up to the mark; the specific gravity of the distillate is then taken with a specific gravity bottle. The contents of the distilling flask are now washed into a 100 c.c. flask, cooled to 60° F. and made up to the mark; the specific gravity is then taken as before; to the specific gravity of the original spirit, add 1000 and deduct the specific gravity of the residue; the difference should be the specific gravity of the distillate, and the percentage of alcohol is obtained by reference to the alcohol tables (see Addenda, p. 213).

Example: The specific gravity of a sample of rum was 953·5; the specific gravity of the distillate was 948·5, and the specific gravity of the residue was 1005·0; then

$$953{\cdot}5 + 1000 = 1953{\cdot}5 - 1005{\cdot}0 = 948{\cdot}5;$$

and by reference to the alcohol tables the rum was 26·20 degrees under proof and contained 35·25 per cent. of alcohol by weight and 42·12 per cent. of alcohol by volume.

Estimation of the total solid matter. 20 c.c. of the sample are placed in a platinum dish and evaporated to dryness on a water bath; the residue is dried in a water oven and cooled and weighed; the weight of the residue multiplied by five gives the total solid matter in percentages.

Example: The weight of the residue obtained from 20 c.c. of a sample of rum was 0·058 gram; then the total solid matter in the sample was $0{\cdot}058 \times 5 = 0{\cdot}29$ per cent.

Calculation of the alcoholic strength from the total solid matter and the specific gravity. One per cent. of total solid matter raises the specific gravity by 3·86; therefore, the specific gravity of the sample, less the total solid matter, multiplied by 3·86, will give the specific gravity from which to obtain the alcoholic strength.

Example: The specific gravity of a sample of rum, containing 0·29 per cent. of total solid matter, was 946·81; then $946{\cdot}81 - (0{\cdot}29 \times 3{\cdot}86) = 945{\cdot}7$; and by a reference to the alcohol tables, the sample was 23·12 degrees under proof, and contained 36·82 per cent. of alcohol by weight, and 43·87 per cent. of alcohol by volume.

Estimation of the percentage of the excess of water. In order to obtain the percentage of the excess of water by volume in a sample of whiskey, rum or brandy, multiply the number of degrees under proof over 25 by 4/3; and in the case of gin, the number of degrees under proof over 35 by 1·54.

Example: A sample of whiskey was 37 degrees under proof, then the percentage of the excess of water by volume in the sample was $(37 - 25) \times 4/3 = 16$ per cent. A sample of gin was 66 degrees under proof, then the percentage of the excess of water by volume in the sample was $(66 - 35) \times 1{\cdot}54 = 47{\cdot}74$ per cent.

The amount by weight of alcohol in percentages is obtained by the use of the formula

$$\frac{\text{percentage by volume} \times 0{\cdot}7938}{\text{specific gravity}};$$

and therefore the percentage volume can be obtained by the formula

$$\frac{\text{percentage by weight} \times \text{specific gravity}}{0{\cdot}7938}.$$

The percentage of proof spirit may be obtained from the percentage of alcohol by volume × 1·75.

Beer. This is a fermented extract of malt and hops. About

35,000,000 barrels of beer are now produced in the United Kingdom annually. Since the middle of the last century beer has been made on scientific principles; the malt is produced by steeping barley in hard water for from three to five days; the water is then drained off and the barley allowed to germinate; it is then transferred to a kiln and dried; during the process the diastase is developed. In the production of beer the malt is crushed between rollers and then mixed with water at a suitable temperature for about two hours, during which time the diastase acts on the starch forming maltose and other carbohydrates; the extract is then drawn off and the residue "sparged" with water at a suitable temperature to wash out the last traces of extract; the extract or "wort" is then passed to the copper where it is boiled and the hops are added; in the copper the diastase is destroyed and the wort concentrated, sterilized and ærated; the wort now passes to the hop back, where the hops are allowed to settle and act as a filter; after about 20 minutes the extract is drained off and passes to the coolers, where albuminous matter is deposited; from here it passes to the fermenting tuns, where the yeast is added, which acts on the maltose and other carbohydrates to form alcohol and carbon dioxide; when the fermentation is complete the yeast is removed and the fermented extract is run into barrels, where the fining materials of isinglass, etc. are added, which remove all suspended matter and give a clear, bright, finished product.

The principal adulterations of beer are the use of hop substitutes, the addition of excessive quantities of salt, and, where invert sugar has been used to prime the beer, the presence of excessive quantities of arsenious oxide, which may be dissolved in the impure sulphuric acid used for the inversion. The salt in beer should not exceed 50 grains per gallon, and the arsenic 1/100th of a grain per gallon.

Estimation of salt. 25 c.c. of the beer are evaporated to dryness in a platinum dish, and carefully incinerated at a low temperature; the residue is extracted with hot water, filtered, and well washed on the filter paper with hot water; the filtrate is cooled and a few drops of potassium chromate

solution are added, followed by N/10 silver nitrate from a burette until a permanent reddish colour is obtained. Each c.c. of N/10 silver nitrate solution used = 0·00585 gram of sodium chloride.

Example: 25 c.c. of beer treated as described required 4·6 c.c. of N/10 silver nitrate to produce a permanent red colour, then 0·00585 × 4·6 = 0·10764 × 700 = 75·35 grains of salt per gallon.

Estimation of arsenic. About 5 grams of arsenic-free zinc are placed in Marsh's arsenic apparatus; a little distilled water and a few drops of arsenic-free hydrochloric acid added and the whole allowed to stand for a few minutes; a small quantity of solid cadmium sulphate is then added; after 15 minutes, during which time the cadmium becomes deposited on the zinc, the water is poured off and the zinc covered with arsenic-free hydrochloric acid; the top of the apparatus is fitted on and the reaction allowed to proceed for 15 minutes; the hydrogen gas, escaping from the end of the arsenic tube, and a small gas flame, so situated that it plays on the neck of the arsenic tube, are lighted; 10 c.c. of the beer are now added, together with a little amyl alcohol and after two or three minutes a small quantity of stannous chloride; the reaction is allowed to proceed for 15 minutes and the deposit is compared with standard deposits from known quantities of arsenic. Care must be taken that the reagents used themselves give no deposit of arsenic.

Detection of hop substitutes. The most convenient method for the detection of hop substitutes is that proposed by Chapman (*Analyst*, 1900, XXV, 36). The method is to take 500 c.c. of the sample and evaporate on a water bath in a porcelain dish; recently ignited sand is added towards the end of the evaporation with constant stirring; the residue is dried in the air oven and finally powdered, and extracted with ether in a stoppered bottle; the ether extract is filtered into a flask and evaporated; the residue is carefully oxidized with an alkaline solution of potassium permanganate (containing 40 grams of potassium permanganate and 10 grams of potassium hydroxide per 1000 c.c.) with vigorous shaking; the excess of potassium permanganate

is reduced with a few drops of a hot solution of oxalic acid; the colourless solution is filtered and evaporated to dryness in a glass dish; the dry residue is acidified with dilute sulphuric acid, when, in the case of genuine hops, the odour of valeric acid is obtained, and in the presence of quassia, acetic acid is liberated; camomile gives the odour of valeric acid, but chiretta does not.

Estimation of sulphites. 25 c.c. of the sample are diluted with 100 c.c. of water; 2 grams of phosphoric acid are added and the mixture distilled; about 80 c.c. of the distillate are collected in bromine water, and heated on a sand bath until all the bromine has been expelled; the solution is then acidified with hydrochloric acid, and barium chloride solution is added; the precipitate of barium sulphate is allowed to settle and is then filtered, well washed, dried in the water oven, incinerated and weighed. The weight of barium sulphate obtained multiplied by 0·2747 gives the weight of sulphite (calculated as SO_2) in 25 c.c. of the beer.

Example: The weight of barium sulphate obtained from 25 c.c. of beer was 0·041 gram; then

$$0{\cdot}041 \times 0{\cdot}2747 = 0{\cdot}0112627 \times 4 = 0{\cdot}045 \text{ per cent.}$$

of sulphite (calculated as SO_2).

Estimation of alcohol in beer. Take 300 c.c. of the beer; place in a distilling flask; add a spoonful of tannic acid to stop the frothing when it is boiled; distil about 200 c.c. and make up the volume to the original 300 c.c.; mix well and take the specific gravity of this distillate in a specific gravity bottle at 15° C. From the specific gravity read off the amount of alcohol from the alcohol tables (p. 213). In the lighter beers the alcohol is about 3 to 4 per cent. and in the heavier beers and porters about 5 to 8 per cent.

Cider. This is an alcoholic beverage produced by the fermentation of apple juice. Perry is a similar beverage produced by the fermentation of pear juice. Cider contains from 2 to 8 per cent. of alcohol. In this country there is no standard for either cider or perry, but both should certainly be alcoholic liquors, and the sale of spurious cider, consisting of a coloured and ærated solution of cane sugar, should be

considered an offence under the Sale of Foods and Drugs Act. The sale of spurious ciders was very common in some seaside resorts, but it has now almost ceased owing to the action of the local authorities. Numerous attempts have been made to suggest chemical standards for cider, but so far none of them are satisfactory.

Detection of spurious cider (Russell and Barker, *Analyst*, 1909, XXXIV, p. 125). 100 c.c. of the sample are evaporated to about 10 c.c. on a water bath; the residue is shaken in a test-tube with an equal volume of ethyl acetate for five minutes and the two liquids are allowed to separate; the ethyl acetate is then drawn off and carefully run down the side of a test-tube on to the surface of a small quantity of lime water; in the presence of a genuine cider, a yellow band is produced at the junction of the two liquids by the action of the lime water on the tannins of the apple juice.

Some Typical Analyses.

	(1)	(2)	(3)
Specific gravity	1025·0	1007·0	1010·0
Acid per cent.	0·45	·034	0·466
Tannin per cent.	0·19	0·18	—
Alcohol per cent.	4·20	5·07	3·64
Total solid matter per cent.	7·85	3·88	4·50
Ash per cent.	0·368	0·322	0·30
Alkalinity of the ash	0·10	0·108	—
Phosphate per cent.	0·20	0·0191	—

Nos. (1) and (2) are analyses of genuine ciders by Russell and Barker (*Analyst, loc. cit.*) and No. (3) is an analysis of a sample of perry by Embrey (*Analyst*, 1891, XVI, p. 41).

CHAPTER IX

VINEGAR—LIME AND LEMON JUICE

VINEGAR.

Vinegar has been defined (*Local Government Board's Annual Report for* 1911–12) as "a liquid derived wholly from alcoholic and acetous fermentations; it shall contain not less than 4 grams of acetic acid (CH_3COOH) in 100 cubic centimetres of vinegar; it shall not contain arsenic in amounts exceeding 0·0143 milligram per 100 c.c. of vinegar; nor any sulphuric acid or other mineral acid, lead or copper; nor shall it contain any foreign substance or colouring matter except caramel. Malt vinegar is derived wholly from malted barley or wholly from cereals, the starch of which has been saccharified by the diastase of malt. Artificial vinegar is any vinegar or substitute for vinegar containing, or derived from any preparation containing, any added acetic acid which is not wholly the product of alcoholic and subsequent acetous fermentation; it shall contain not less than 4 grams of acetic acid (CH_3COOH) in 100 c.c. of artificial vinegar; it shall not contain arsenic in amounts exceeding 0·0143 milligram per 100 c.c. of artificial vinegar, nor any sulphuric acid or other mineral acid, lead or copper, nor shall it contain any foreign substance or colouring matter except caramel."

There are two chief varieties of vinegar sold in this country, malt vinegar and "wood" vinegar. "Wood" vinegar is obtained by diluting concentrated acetic acid with water and adding a little caramel as colouring matter. Malt vinegar should be obtained from malted barley or from a mixture of malted and unmalted barley; the malt or the mixture is ground and mashed with successive quantities of hot water, which is then run off and treated with yeast; this causes fermentation with production of alcohol, which is subsequently oxidized to acetic acid by passing the wort over birch twigs, which have been impregnated with *Mycoderma aceti*. Malt

vinegar contains traces of alcohol, gum, sugar, extractive, acetates, ethers, aldehyde, phosphates, etc. and is usually sold at 18, 20, 22 or 24, the last contains 6 per cent. of acetic acid and has a specific gravity of 1019; the figures denote that one fluid ounce will neutralize 18, 20, 22 or 24 grains respectively of pure dry sodium carbonate.

Malt vinegar is usually adulterated by the partial or total substitution of "wood" vinegar or the addition of mineral acid. The detection of "wood" vinegar in malt vinegar was formerly possible by the determination of the nitrogen figure, but of late years manufacturers of malt vinegar, in order to keep the vinegar, have removed a portion of the nitrogen by precipitation and this figure has in consequence become of no practical importance. The presence of artificial vinegar is now detected by the amount of phosphate present; the percentage of phosphate in genuine malt vinegar should not be less than 0·05 per cent. calculated as P_2O_5 (Russell and Hodgson, *Analyst*, 1910, XXXV, 346).

Determination of the total solid matter. Evaporate 25 c.c. of the vinegar in a platinum dish and dry the residue in a water oven until constant; the weight of the residue multiplied by four gives the percentage of the total solid matter per 100 c.c.

Example: The residue from 25 c.c. of a sample of vinegar weighed 0·586 gram; then $0·586 \times 4 = 2·344$ gram of total solid matter per 100 c.c.

Ash. The residue obtained above is carefully incinerated over a Bunsen flame until a white ash is obtained; the weight of this ash multiplied by four gives the weight of ash per 100 c.c.

Example: The weight of ash obtained from 25 c.c. of vinegar was 0·078, then $0·078 \times 4 = 0·312$ gram of ash per 100 c.c.

Estimation of acetic acid. Dilute 10 c.c. of the vinegar to about 200 c.c., add a few drops of phenol phthalein solution and titrate with N/1 sodium hydroxide solution until a permanent pink colour is obtained; each c.c. of the N/1 sodium hydroxide solution used = 0·06 gram of acetic acid.

Example: 10 c.c. of a sample of vinegar required 10·3 c.c.

of N/1 sodium hydroxide solution to produce a permanent pink colour; then $10{\cdot}3 \times 0{\cdot}06 \times 10 = 6{\cdot}18$ gram of acetic acid per 100 c.c.

Calculation of original solids. Theoretically 60 grams of acetic acid are produced from 90 grams of glucose; therefore the acetic acid found multiplied by 1·5 gives the amount of sugar from which the acetic acid was derived; to this is added the amount of total solid matter contained in the vinegar, giving the original solid matter.

Example: A sample of vinegar contained 4·8 grams of acetic acid and 2·34 grams of total solid matter per 100 c.c., then the original solids were $(4{\cdot}8 \times 1{\cdot}5) + 2{\cdot}34 = 7{\cdot}20$ grams per 100 c.c.

Estimation of phosphate. Evaporate 50 c.c. of the vinegar to dryness; incinerate the residue and dissolve the ash in dilute nitric acid; a few c.c. of a solution of ammonium molybdate are added and the mixture is kept at 60° C. for two hours; in the presence of phosphate the characteristic canary yellow precipitate is obtained; this is filtered off and washed with water containing ammonium molybdate; the washed precipitate is then dissolved off the filter paper with dilute ammonia, and a few c.c. of a cold magnesia mixture added; the mixture is allowed to stand for 12 hours; the precipitate is then filtered off, washed with dilute ammonia water, dried in the water oven, incinerated, ignited over a blowpipe and weighed as magnesium pyrophosphate ($Mg_2P_2O_7$); the weight of magnesium pyrophosphate obtained multiplied by 0·6396 and then multiplied by two gives the weight of phosphate (as P_2O_5) in 100 c.c. of the vinegar.

Example: 50 c.c. of vinegar gave 0·0295 gram of magnesium pyrophosphate then $0{\cdot}0295 \times 0{\cdot}6396 \times 2 = 0{\cdot}038$ gram P_2O_5 per 100 c.c.

Detection of free mineral acid. When acetates and other salts of organic acids are incinerated, alkaline carbonates are formed; but when the salts of mineral acids are ignited the residue has a neutral reaction; therefore, if the ash of a vinegar is alkaline, mineral acids are not present; but if the ash is neutral, mineral acids have probably been added.

Estimation of free mineral acid. Qualitative tests for chlorides and sulphates are first performed and 25 c.c. of vinegar are placed in a platinum dish with 20 c.c. of N/10 sodium hydroxide solution; the whole is evaporated to dryness and incinerated; the ash is dissolved in water and a few drops of methyl orange are added; N/10 sulphuric acid is then added until a permanent pink colour is obtained; in the absence of mineral acids 20 c.c. of N/10 sulphuric acid would be used; the amount of mineral acid present is calculated from the difference between the acid used and 20 c.c.

Example: A sample of vinegar treated as described above required 15 c.c. of N/10 sulphuric acid to produce a permanent pink colour; qualitative tests showed sulphates to be present; then $20 - 15 = 5$; each c.c. represents 0·0049 gram of sulphuric acid or 0·00365 gram of hydrochloric acid, therefore the sample contained $5 \times 0{\cdot}0049 = 0{\cdot}0245 \times 4 = 0{\cdot}098$ gram of H_2SO_4 per 100 c.c.

Typical Analyses of Vinegar (in percentages).

	(1)	(2)	(3)	(4)	(5)
Total solid matter (grs. per 100 c.c.)	2·70	1·47	2·36	0·472	1·54
Ash (grs. per 100 c.c.)	0·60	0·42	0·23	0·041	0·20
Alkalinity of ash (as K_2O) (grs. per 100 c.c.)	0·032	0·037	0·035	0 014	0·07
Phosphates (as P_2O_5) (grs. per 100 c.c.)	0·051	0·078	0·020	nil	0·005
Acetic acid (grs. per 100 c.c.)	4·80	6·15	4·80	2·20	3·50
Specific gravity	1019·3	1018·0	1017·0	1012·1	1010·0

Nos. (1) and (2) are genuine malt vinegars; No. (3) is a malt vinegar admittedly "made without the use of malt"; No. (4) is a "wood" vinegar; and No. (5) is a sugar vinegar.

LIME AND LEMON JUICE.

Lime juice is the expressed juice of *Citrus medica acida*, and lemon juice of *Citrus medica limonum*. According to the official description of lime juice in the British Pharmacopœia it should contain from 7 to 9 per cent. of citric acid and should have a specific gravity at 60° F. of 1030 to 1040; it should

contain about 14 per cent. of total solids and the ash should not exceed 3 per cent. of the total solids. Commercially these syrups are sold as lime juice cordial and lemon squash in order that they may not be subject to the provisions of the Sale of Foods and Drugs Acts, and they usually contain from 2 to 4 per cent. of citric acid, and from 30 to 40 per cent. of total solid matter, the latter consisting chiefly of added sugar, but it is doubtful whether or not the sale of such substances are offences under the act.

Estimation of free citric acid. Dilute 10 c.c. of the lime juice to about 100 c.c. and titrate with N/10 sodium hydroxide solution using phenol phthalein as an indicator; each c.c. of N/10 sodium hydroxide solution used represents 0·007 gram of citric acid.

Example: 10 c.c. of a sample of lime juice required 44·3 c.c. of N/10 sodium hydroxide solution to produce a permanent pink colour; then $44{\cdot}3 \times 0{\cdot}007 \times 10 = 3{\cdot}10$ per cent. of citric acid.

Estimation of combined citric acid. Exactly neutralize 10 c.c. of the lime juice with N/10 sodium hydroxide solution and evaporate the neutral solution to dryness on a water bath; incinerate the residue over a Bunsen flame and extract the ash with hot water; to the water extract add 20 c.c. of N/1 sulphuric acid and titrate with N/1 sodium hydroxide solution, using methyl orange as an indicator, until an orange colour is obtained; from the number of c.c. of N/1 sulphuric acid used deduct the number of c.c. of N/1 sodium hydroxide solution required and multiply the difference by 0·07; this gives the total citric acid in 10 c.c. of the lime juice.

Example: 10 c.c. of a sample of lime juice treated as above required 13·7 c.c. of N/1 sodium hydroxide solution to produce an orange colour; then $20{\cdot}0 - 13{\cdot}7 = 6{\cdot}3$ c.c. of N/1 sulphuric acid used; $6{\cdot}3 \times 0{\cdot}07 \times 10 = 4{\cdot}41$ per cent. of total citric acid; the sample contained 3·08 per cent. of free citric acid; therefore the sample contained $4{\cdot}41 - 3{\cdot}08 = 1{\cdot}33$ per cent. of combined citric acid.

Mineral acid. The addition of mineral acid to lime juice has become common, the manufacturers' view being that it

is necessary to prevent decomposition. The presence of mineral acid is detected and the amount estimated as described under vinegar (pp. 163, 164).

Preservatives. Most samples of lime juice cordial and lemon squash which do not contain mineral acid, contain instead some other preservative; the most common being salicylic acid and sulphite; the methods for the estimation of these substances will be given later (p. 171).

Typical Analyses of Lime Juice (in percentages).

	(1)	(2)
Total solid matter ..	12·56	6·65
Free citric acid	7·33	3·08
Combined citric acid ..	1·41	1·33
Ash	0·16	0·20
Sulphite	—	0·06

No. (1) is a genuine lime juice; No. (2) is deficient in solids and citric acid and contains sulphite preservative.

CHAPTER X

POISONOUS METALS IN FOODS

The most common poisonous metals found in foods are tin, copper and arsenic. Tin appears in canned foods owing to the action of the acid juices, or owing to the action of putrefactive gases on the container. The proportion of tin should not exceed 2 grains per pound (*Local Government Board Food Reports*, No. 7). Copper is used in the so-called "greening" of preserved vegetables with the object of bringing out the deep green colour especially of preserved peas. The Departmental Committee on Preservatives and Colouring Matters in Food, 1901, reported that "the use of copper salts in the so-called greening of preserved foods be prohibited." Arsenic appears in beer from the use of impure sugar, and also in a number of other foods. The Royal Commission on Arsenical Poisoning, 1903, reported that "In our view it would be entirely proper that penalties

should be imposed under the Sale of Foods and Drugs Acts upon any vendor of beer or any other liquid food, or of any liquid entering into the composition of food, if that liquid is shown by an adequate test to contain 1/100th of a grain or more of arsenic in the gallon and with regard to solid food—no matter whether it is habitually consumed in large or in small quantities or whether it is taken by itself (like golden syrup) or mixed with water or other substances (like chicory or " carnos ")—if the substance is shown by an adequate test to contain 1/100th grain of arsenic or more in the pound."

Determination of tin in canned foods (Buchanan and Schryver, *Local Government Board Food Reports*, No. 7). *Solutions required:* Stannous chloride solution containing 1·428 grams of stannous chloride per 500 c.c.

Solution of dinitrodiphenylaminesulphoxide containing 0·2 gram in 100 c.c. of N/10 sodium hydroxide solution.

Preparation of dinitrodiphenylaminesulphoxide. Ten parts of nitric acid (specific gravity 1·48) are mixed with 10 parts of nitric acid (specific gravity 1·40) and the mixture cooled with ice and salt; 1 part of thiodiphenylamine is then added in small quantities at a time, with continual stirring; the temperature should not be allowed to rise above 5° C. and such small quantities should be added at a time that the hissing sound is hardly perceptible when the solid matter comes into contact with the liquid mixture. The thiodiphenylamine dissolves at first in the nitric acid to form a clear red solution, which gradually thickens owing to the separation of the nitro body. The solution is allowed to stand for some hours (not more than half a day); the nitro body is then sucked off on an asbestos filter and washed, first with concentrated nitric acid and then with nitric acid of gradually diminishing strength, and finally with pure water; it is then extracted with hot alcohol, in which it is not appreciably soluble.

The process. 50 grams of the meat or fruit are introduced into two 700 c.c. round-bottomed Jena flasks (25 grams in each); 10 grams of powdered potassium sulphate and 30 c.c. of concentrated sulphuric acid are added; the contents of the flasks are then heated till colourless as described in

Kjeldahl's method for the estimation of nitrogen (p. 85). When the flasks are cold, the contents are diluted with water to about 100 c.c. and mixed; sulphuretted hydrogen is then passed through the solution, the mixture is allowed to stand overnight in a well corked flask; the contents of the flask are slightly warmed on a water bath and filtered through a filter paper (4 cm. in diameter); the precipitate is washed with water, and the precipitate and filter paper are transferred to a test-tube and boiled with 5 c.c. of concentrated hydrochloric acid to dissolve the sulphide; the solution is filtered through a small conical Buchner funnel into a wide mouthed test-tube with a side tube near the top, by means of which the test-tube is connected with a filter pump; the filter paper is sucked as dry as possible and washed with 25 c.c. of concentrated hydrochloric acid; the test-tube is then connected with a carbonic acid gas apparatus, and gas is passed through the solution by means of a tube passing through the cork placed in the mouth of the test-tube and reaching down to the surface of the liquid; the side tube serves as an exit for the gas. Whilst the solution is still hot 0·75 gram of zinc foil is thrown into the strongly acid liquid, and, as soon as the last traces of zinc are dissolved, 2 c.c. of the reagent are added, by means of a pipette, to the hot liquid; the cork of the tube is momentarily lifted for the insertion and carbonic acid gas is passed the whole time. On the addition of the reagent, the nitro body is at first precipitated but afterwards redissolves; the solution is then boiled for two minutes and diluted to 100 c.c. with cold water; this diluted solution is filtered with the aid of the filter pump from the unchanged nitro body and usually turns violet during the filtration; a drop of dilute ferric chloride solution is added to bring out the maximum colour, which is then matched with known quantities of the standard tin solution.

Gravimetric estimation. The organic matter is destroyed as described above; and, when the incineration is complete, the contents of the flask are diluted, mixed and further diluted to about 600 c.c.; excess of sulphuretted hydrogen gas is now passed through the solution and the mixture is allowed to stand

overnight in a well corked flask ; the contents of the flask are then slightly warmed, and the mixture of sulphide and sulphur filtered through a filter paper (7 cm. in diameter) ; the precipitate is washed with warm water and dissolved off the filter paper with a small quantity (10 to 20 c.c.) of a hot 10 per cent. solution of sodium hydroxide ; from the yellow solution thus obtained, the sulphide is reprecipitated by means of glacial acetic acid and filtered ; the precipitate is washed with hot water, dried in a water oven, incinerated and weighed as oxide.

Determination of copper in preserved peas. 10 grams of the sample are incinerated in a porcelain dish ; the residue is dissolved in 3 c.c. of concentrated nitric acid ; filtered, and the filtrate diluted to 100 c.c. ; an electric current is then passed through this diluted solution contained in a platinum dish so that the dish is the kathode and the copper is deposited on the platinum dish ; as soon as all the copper is deposited the anode is removed, and the solution in the platinum dish is at once poured off the copper, which is well washed with distilled water, dried in a water oven and weighed.

Example : 10 grams of a sample of peas were incinerated and the residue made up to 100 c.c. and from 50 c.c. of this solution 0·024 gram of copper was deposited ; then

$$0{\cdot}024 \times 20 = 0{\cdot}48 \text{ per cent.}$$

of copper in the sample.

Detection of arsenic. (1) *Reinsch's test.* When a clear, bright piece of copper foil is boiled in a hydrochloric acid solution of an arsenic compound, a grey film appears on the copper ; the foil is gently washed and dried ; placed in a tube of hard glass and gently heated ; crystals of arsenious oxide will appear on the cold parts of the tube ; a blank test should always be carried out with the acid used. This test is used in toxicological examinations and is capable of detecting one part of arsenic in 250,000 parts of solution.

(2) *The electrolytic test.* The apparatus for this test consists of a platinum cathode in a glass cylinder, which is open at one end and fits into a porous cell ; this is surrounded

by an anode and stands in a glass vessel; the upper portion of the cylinder is fitted with a ground glass neck for insertion of the drying tube and funnel; a hard glass capillary tube fits into the end of the drying tube, and is surrounded at the shoulder by a piece of platinum gauze, which can be heated by a small Bunsen flame.

The process. Carefully wash the apparatus with distilled water; fill the outer cell with a 30 per cent. solution of sulphuric acid and partially fill the inner cell; a current of electricity (not exceeding 5 ampères) is then passed through the apparatus, and, as soon as all the air has been expelled, the escaping hydrogen is lighted and also the small Bunsen flame; the current is run for 15 minutes; if no deposit appears in the capillary tube in that time, the materials are free from arsenic. A solution of the food to be tested is made in 30 per cent. sulphuric acid, and passed through the funnel into the inner cell; the current is again turned on, and, after passing for 15 minutes, the capillary tube is fused off at the shoulder and the deposit obtained is compared with standard deposits.

Preparation of the standard deposits. Weigh out 0·1 gram of pure dry finely ground oxide of arsenic (As_2O_3); wash it into a 1000 c.c. flask and make up to the mark. Each c.c. of this solution represents 0·0001 gram of oxide of arsenic. Measure out 100 c.c. of this solution and dilute it to 1000 c.c. Each c.c. of this dilute solution contains 0·00001 gram of oxide of arsenic. Standard deposits of 0·00001 gram, 0·000008 gram, 0·000006 gram, and 0·000004 gram of oxide of arsenic are obtained by placing 1 c.c., 0·8 c.c., 0·6 c.c., and 0·4 c.c. respectively of the dilute arsenic solution in the apparatus, and, after the deposit is obtained, the capillary tube is sealed at both ends; the deposit will keep for some time, but should be renewed occasionally.

(3) *The Gutzeit test.* Goode and Perkin's modification (*Jour. Soc. Chem. Ind.* 1906, xxv, 507). The apparatus consists of a conical flask of 250 c.c. capacity fitted with a dropping funnel and attached to a U-tube, containing a 10 per cent. solution of acid cuprous chloride; the exit from the U-tube is restricted by inserting a rubber bung, through which

passes a glass tube of 5 cm. diameter. About 5 grams of ammonium chloride are first placed in the flask; then 1 to 2 grams of magnesium turnings, and 5 to 10 grams of the food are added, and 10 c.c. of water are run on to the mixture; the apparatus is gently shaken and placed in a basin of cold water. A piece of filter paper, soaked in a strong alcoholic solution of mercuric bromide, is placed over the outlet, and kept there for one hour or, in the presence of arsenates, for one and a quarter hours; the paper is then placed on a watch glass, moistened with a few drops of concentrated hydrochloric acid, warmed and the acid poured off; the paper is then washed with a little water and allowed to dry; in the presence of arsenic a red stain will be produced on the filter paper; it is claimed that by this method 0·000001 gram of arsenic is easily detected.

The detection of poisonous metals in water is described in Chap. 1, pp. 16 and 17.

The reports of the Royal Commission on Arsenical Poisoning, 1903 (Cd. 1869, Cd. 1845, and Cd. 1848), are of importance in connection with arsenic in foods; the final Report, part 1 (Cd. 1848), sums up and discusses the evidence.

CHAPTER XI

PRESERVATIVES—DISINFECTANTS

PRESERVATIVES IN FOODS.

Sulphite preservative is generally present in the form of calcium or sodium bisulphite; it is often found in preserved meat, in beer and wines from the "sulphuring" of the casks. The Departmental Committee's Report on Preservatives and Colouring Matters in Food (1901) made no recommendation as to the use of this preservative in foods or drinks. In the United States of America the Federal rules limit it to 350 milligrams per litre, provided that not more than 70 milligrams

are in a free state. In France the amount permitted in wines and ciders is limited to 100 milligrams of the acid per litre whether "free" or "combined." In Austria-Hungary it is limited to 80 milligrams per litre. Durham (*Jour. of Hygiene*, 1909, vol. XVII, p. 17) suggests that in cider and perry more than a trace of sulphite should be labelled with a declaration to that effect; and that the word "trace" should be defined as "less than 10 (or possibly) 20 milligrams of SO_2 per litre."

Estimation of sulphite. 50 grams of the sample, or, in the case of a liquid, 50 c.c., are made into a paste with water and transferred to a distilling flask; the mixture is acidified with phosphoric acid and the distillate collected in a large excess of N/10 iodine solution; the excess of N/10 iodine solution is then titrated with N/10 sodium thiosulphate solution; using starch solution as an indicator. Each c.c. of N/10 iodine solution used = 0·0032 gram of sulphite, calculated as sulphur dioxide. The presence of more than 0·03 per cent. of sulphite must be regarded as an adulteration.

Example: In an experiment carried out as described above, 50 grams of meat gave sufficient sulphur dioxide to neutralize 32 c.c. of N/10 iodine solution; then, $32 \times 0{\cdot}0032 \times 2 = 0{\cdot}205$ per cent. of sulphite (calculated as sulphur dioxide).

Salicylic acid is sometimes added to beer and very often to British wines, lime and lemon juice. The most convenient method for the estimation is that proposed by Harry and Mummery (*Analyst*, 1905, XXX, 124).

The process. 100 c.c. of the sample are placed in a 300 c.c. flask, and made alkaline with N/1 sodium hydroxide solution; the mixture is evaporated until all the alcohol has been driven off; the residue is then cooled and neutralized with N/1 sulphuric acid; at least 20 c.c. of a saturated solution of lead sub-acetate are then added; the whole is made alkaline with 20 c.c. N/1 sodium hydroxide solution; made up to the mark with water and filtered; 100 c.c. of the filtrate are made acid with hydrochloric acid; the precipitate of lead chloride is filtered off and the filtrate is extracted three times with ether; the mixed ethereal residues are evaporated, and the residue

dissolved in dilute alcohol, washed into a Nessler cylinder and made up to the mark; the salicylic acid is then estimated colourimetrically as described under Jam (p. 152).

Estimation of boric acid in meat. Fresenius and Popp (*Chem. Centr.* 1897, II, 69). The meat is finely chopped and 10 grams are well mixed with 40 to 80 grams of anhydrous sodium sulphate; dried in a water oven; and, when perfectly dry, finely powdered and washed into a 300 c.c. conical flask with 100 c.c. of methyl alcohol; the mixture is allowed to stand for 12 hours; the alcohol is then distilled off, and the residue is washed with a further quantity of 50 c.c. of methyl alcohol, which is also distilled off; the mixed distillates are made up to 150 c.c. with methyl alcohol, and to 50 c.c. are added 50 c.c. of water and 50 c.c. of a 50 per cent. solution of glycerine and a few drops of phenol phthalein solution; the solution is then titrated with N/10 sodium hydroxide solution until a permanent pink colour is obtained. Each c.c. of N/10 sodium hydroxide solution used corresponds to 0·0062 gram of boric acid.

Detection of benzoic acid. *In milk, cream, and butter.* Hinks (*Analyst*, 1913, XXXVIII, 555). For a qualitative test 25 c.c. of milk or 10 to 20 grams of cream or butter are heated with an equal volume of concentrated hydrochloric acid (as in the Werner-Schmidt method of estimating fat in milk, p. 83) and cooled; the solution is extracted with 20 c.c. of a mixture of ether (1 vol.) and petroleum ether (2 vols.); the ethereal solution is separated, and one drop of ammonia solution and 5 c.c. of water are added to it; the mixture is shaken and the aqueous layer separated; the ammonia is expelled by heating on a water bath, and a few drops of a neutral solution of ferric chloride are added; in the presence of benzoic acid a buff coloured precipitate is obtained.

For a quantitative test the same volume of milk or weight of cream as above is heated with hydrochloric acid under a reflux condenser, cooled and extracted three times with 20 c.c. of the ether mixture; the mixed ethereal extracts are made alkaline with ammonia, and 10 c.c. of water are added; the mixture is well shaken, the aqueous layer is separated and

acidified with hydrochloric acid and extracted with the mixed ether as before; the ethereal extract is allowed to evaporate spontaneously and the residue is dried to a constant weight in a desiccator; the weighed residue is placed in an air oven at 100° C. for two hours, cooled and reweighed; the loss in weight represents benzoic acid. Boric acid does not interfere with the reaction.

In meats. Fischer and Gruenert (*Zeit. Untersuch. Nahr. Genussm.* 1910, XX, 580). 50 grams of the finely divided meat are shaken with 150 c.c. of a 1 per cent. solution of sodium bicarbonate at the melting point of the fat; the hot aqueous solution is separated and neutralized with dilute sulphuric acid; 10 c.c. of Fehling's copper solution, and 10 c.c. of a 3·1 per cent. solution of potassium hydroxide are added and the mixture is filtered; the filtrate is acidified with dilute sulphuric acid and extracted with ether; the ether extract is evaporated at a low temperature; the residue is dissolved in water containing ammonia and evaporated to 1 c.c.; and one drop of a dilute neutral solution of ferric chloride is added; in the presence of benzoic acid a buff coloured precipitate is obtained.

Detection of fluorides in butter and margarine. Monier-Williams (*Local Government Board Food Reports*, No. 17). Advantage is taken of the bleaching action of fluorides on peroxidized titanium solution. 10 grams of the butter or margarine are melted and shaken in a separating funnel with ether and a small quantity of water; the water layer is run off into a test-tube; a few drops of hydrogen peroxide are added and 1 c.c. of a 2 per cent. solution of titanium sulphate in a 10 per cent. solution of sulphuric acid solution; in the presence of fluorides the yellow colour of the titanium solution is partially discharged, a fact which is easily seen when the solution is compared with that obtained at the same time from genuine butter.

Estimation of formaldehyde in meats, etc. Romijn (*Zeit. Anal. Chem.* 1897, XXXVI, 18). To 100 grams of the finely chopped meat 100 c.c. of water and 1 c.c. of sulphuric acid (1 in 3) are added in a distillation flask; the mixture is

carefully distilled over a low flame until 20 c.c. of distillate have been collected; this distillate contains one-third of the total formaldehyde present; to the distillate (which should not contain more than 0·3 per cent. of formaldehyde) are added 25 c.c. of N/10 iodine solution in a stoppered flask; a dilute solution of potassium hydroxide is then added drop by drop until the solution is yellow; the mixture is allowed to stand for 15 minutes; concentrated hydrochloric acid is then added until the red colour is restored, and the excess of iodine is titrated with N/10 sodium thiosulphate solution. The weight of iodine absorbed multiplied by 0·118 gives the weight of formaldehyde in the distillate, and this multiplied by three gives the weight (per cent.) of formaldehyde.

The detection of various preservatives in milk and cream is described in Chap. II, pp. 92 and 100.

The Report of the Departmental Committee on the use of Preservatives and Colouring Matters in the preservation and colouring matters of food, 1901 (Cd. 833), contains the minutes of the evidence taken before the Committee as well as a full discussion.

DISINFECTANTS.

A disinfectant is a solid or a liquid which is used for the purpose of destroying injurious organisms, and is, generally, though not necessarily, an antiseptic and deodorant. The actual germ-killing power of a disinfectant cannot be determined by chemical means, but the proportion of the active substance can be determined. A large number of commercial disinfectants are of little or no practical value for the purpose for which they are intended, and local authorities are largely to blame for the rubbish which is supplied to them as disinfectants, as it is a common practice to advertise for tenders for the supply of disinfectants "containing 10 per cent. or more of liquid carbolic acid." The local authority expects to be supplied with a disinfectant containing 10 per cent. or more of absolute phenol, the price of which would of course be absolutely prohibitive. The methods of analysis of only a few of the more important disinfectants are described here.

Estimation of hydrogen peroxide. The amount of hydrogen peroxide can be stated as the amount of "available" oxygen either by volume or by weight. A solution of the substance containing 3 per cent. by weight of H_2O_2 corresponds to a solution which liberates 10 times its volume of available oxygen. And 100 c.c. of such a solution which gives off 1000 c.c. of oxygen at 0° C. and 760 mm. is called a 10-volume solution.

The method of estimation is to add 5 c.c. of dilute sulphuric acid to about 500 c.c. of water in a dish; N/10 potassium permanganate is run in until the faint pink colour is permanent; now add 5 c.c. of the hydrogen peroxide solution and run in the permanganate solution till the faint pink colour becomes permanent. If the first drop of permanganate causes a permanent colouration add a little more sulphuric acid; and if the colour still persists for a few minutes it means that no hydrogen peroxide is in solution. The equation representing the change is of course

$$5H_2O_2 + 2KMnO_4 + 4H_2SO_4 = 2MnSO_4 + 2KHSO_4 + 8H_2O + 5O_2.$$

Hydrogen peroxide is usually of three different strengths, and contains 5, 10, or 20 times its own volume of "available" oxygen. The "ten-volume" solution gives off ten times its volume of oxygen, and this is equivalent to 3·04 per cent. of H_2O_2, and to 1·43 per cent. by weight of "available" oxygen. It is used in medicine, and for killing pathogenic and other organisms in milk and other foods. It is generally used in the form of a 1 per cent. solution.

Sulphur dioxide destroys vermin but not spores; the dry gas has little or no effect and it must be used in a moist condition. It is generally obtained by burning sulphur candles and is used to disinfect verminous houses, for sterilizing beer barrels, and, in the form of bisulphite, as a food preservative.

Estimation of available sulphur dioxide. Muter (*Analyst*, 1890, xv, 63). 2 grams of the powder are placed on a filter and washed with anhydrous ether to remove tarry matters; the residual ether is evaporated from the powder, which is then added to 50 c.c. of N/10 iodine solution and allowed to stand for 30 minutes with frequent shaking; the contents of the bottle

are then titrated with N/10 sodium thiosulphate solution, using starch solution as an indicator. Each c.c. of iodine used represents 0·0032 gram of available sulphur dioxide.

Sulphite disinfectants oxidize rapidly, and if kept badly may depreciate very rapidly; it is necessary therefore to estimate this "reversion."

Estimation of the "reverted" sulphur dioxide. Muter (*Analyst*, 1890, XV, 63). 20 grams of the sample are placed in a bottle and 200 c.c. of water are added; the mixture is shaken occasionally, allowed to settle and filtered; 20 c.c. of the filtrate (= 2 grams of the sample) are mixed with an excess of bromine and filtered; the filtrate is treated with an excess of barium chloride solution and the precipitate is collected on a filter paper, dried in a water oven, incinerated and weighed; the weight of barium sulphate obtained multiplied by 0·2747 gives the amount of total sulphur dioxide in 2 grams of the sample; the available sulphur dioxide deducted from this gives the "reverted" sulphur dioxide.

Lime and chloride of lime are excellent disinfectants for large quantities of material; they kill bacteria but not spores, and are used for the disinfection of enteric fever and cholera discharges. Chloride of lime depends for its activity on the liberation of chlorine, and it is therefore necessary to know the proportion of available chlorine in a sample.

Estimation of available chlorine in chloride of lime. Penot's method (*J. Prakt. Chem.* 1896, LIV, 59). *Solutions required: N/10 sodium arsenite solution.* Weigh out 4·95 grams of pure powdered arsenious oxide and dissolve in 250 c.c. of water; 20 grams of sodium carbonate are added and the whole warmed and shaken until solution is complete, and finally diluted to 1000 c.c.

N/10 iodine solution. Weigh out 12·7 grams of pure resublimed iodine, and 18 grams of pure potassium iodide, and dissolve them together in 250 c.c. of water; dilute the solution to 1000 c.c.

The process. Weigh out 7·09 grams of the chloride of lime, mix it with water in a mortar until a smooth cream is formed; wash the cream into a 1000 c.c. flask and dilute it to 1000 c.c.;

to 50 c.c. of this solution (= 0·3545 gram of the powder), add excess of the sodium arsenite solution ; the excess of sodium arsenite is then determined by titration with the N/10 iodine solution, using starch solution as an indicator, until a permanent blue colour is obtained. Each c.c. of the sodium arsenite solution used represents 1 per cent. of available chlorine.

Pontius' method (*Chem. Zeit.*, 1904, XXVIII, 54). *Solution required: N/10 potassium iodide solution.* Weigh out 16·6 grams of pure potassium iodide, dissolve it in water, and dilute the solution to 1000 c.c.

The process. Dissolve 7·09 grams of the chloride of lime in 1000 c.c. of water as above ; to 50 c.c. of this solution (= 0·3545 gram of the powder), add 3 grams of solid sodium carbonate, and 2 to 3 c.c. of starch solution ; this solution is immediately titrated with the N/10 potassium iodide solution until the blue colour is permanent. Each c.c. of N/10 potassium iodide solution used represents 0·0213 gram of available chlorine.

This method is very convenient and rapid and is applicable to the determination of available chlorine in powders of unknown strength, but the results are only approximate.

Permanganates of potassium and sodium must always be used in excess ; the presence of excess of the permanganate is shown by the permanent pink colour; they are excellent deodorisers but are very costly and occasionally imperfect. " Condy's fluid " is a solution of sodium permanganate.

The estimation of the strength of a solution of potassium permanganate depends upon the following chemical changes :

$$2KMnO_4 + 5C_2H_2O_4 . 2H_2O + 3H_2SO_4 = 10CO_2 + K_2SO_4 + 2MnSO_4 + 18H_2SO.$$

A N/10 solution of the oxalic acid is made by dissolving 6·301 grams of the pure crystals in one litre of pure water.

50 c.c. of the oxalic acid solution are placed in a beaker and a few c.c. of sulphuric acid added together with about 300 c.c. of water ; warm to about 60° C. and the solution of potassium permanganate run in till the faint pink tint remains permanent. The strength of the solution can then be calculated from the above equation.

Mercuric chloride. "Corrosive sublimate" is one of the most powerful disinfectants; it should not be used for the disinfection of sputum or excreta or with hard water or sulphides.

Formaldehyde or "formalin" is used in the form of a spray and has the advantage that it is easily applied; it is very effective and may be used with practically anything.

Estimation of formaldehyde. The commercial "formalin" should contain about 40 per cent. of formaldehyde. It is estimated by oxidizing it to formic acid by iodine in an alkaline solution according to the following equation:

$$H . COH + H_2O + I_2 = 2HI + H . COOH.$$

10 c.c. of the solution are diluted to 400 c.c. with pure water; then place 10 c.c. of this 1 per cent. solution in a beaker and mix with 100 c.c. N/10 iodine; add sodium hydroxide drop by drop till the colour becomes bright yellow; allow to stand for about 10 minutes, acidify with dilute hydrochloric acid, and titrate the liberated iodine with N/10 sodium thiosulphate. Each c.c. of the N/10 iodine which disappears when the formaldehyde is oxidized to formic acid = 0·0015 gram of formaldehyde.

Coal tar disinfectants rely for their effectiveness chiefly on the proportion of phenol, and its allies, which they contain. They are in the form of liquids and powders, and occasionally contain little or no phenol, but comparatively large quantities of tar oils.

Estimation of tar oils. Muter (*Analyst*, 1890, xv, 63). 50 grams of the powder, or 50 c.c. of a liquid, are shaken with 200 c.c. of a 10 per cent. solution of sodium hydroxide and the bottom layer run off; the upper layer is washed with a 5 per cent. solution of sodium hydroxide and the whole is rapidly filtered; the precipitate on the filter paper is washed into a beaker with water and filtered again through a weighed filter paper; the filter paper is cautiously dried between blotting paper, placed in a desiccator for 12 hours and weighed; the increase in weight represents tar oils.

Estimation of phenol. *In a powder containing no lime.* 50 grams of the sample are placed in a Soxhlet apparatus and

completely extracted with ether ; the ethereal solution is shaken twice with 20 c.c. of a 20 per cent. solution of sodium hydroxide ; the alkaline solution is then evaporated to about 10 c.c., poured into a burette, acidified with dilute sulphuric acid and allowed to stand till perfectly cold ; a layer of phenol separates and its volume is read off in c.c. The number of c.c. of phenol obtained multiplied by 1·05 gives the number of grams of phenol in the 50 grams of powder taken.

In a powder containing lime. 50 grams of the sample and 5 c.c. of water are placed in a large mortar ; dilute sulphuric acid (50 per cent. by volume) is then added a few drops at a time until the solution is acid to litmus ; this addition should take several hours and the temperature should not be allowed to rise, otherwise some of the phenol will be lost by evaporation ; the residue is then placed in a Soxhlet apparatus and treated as described above.

In liquid preparations. 100 c.c., or less, according to the supposed strength of the sample, are placed in a litre distilling flask with 150 c.c. of distilled water, acidified with dilute sulphuric acid, and distilled ; 100 c.c. of water are then added and the mixture is again distilled ; the mixed distillates are made up to 500 c.c. and the phenol determined by Wilkie's method (*Jour. Soc. Chem. Ind.* 1911, xxx, 398). To 100 c.c. of the distillate are added equal volumes of N/10 sodium carbonate solution and N/10 iodine solution ; after standing for 5 minutes, the sodium carbonate is neutralized with N/10 sulphuric acid, and the excess of iodine is titrated with N/10 sodium thiosulphate solution, using starch solution as an indicator. Each c.c. of N/10 iodine solution absorbed = 0·0127 × 0·1235 gram of phenol.

Koppeschaar's method of estimating the amount of phenol. Shake up bromine with water until the latter is saturated ; 0·25 gram of pure dry crystalline carbolic acid is weighed ; place in a well-stoppered flask ; dissolve in 100 c.c. of distilled water ; run in the bromine water from a burette, with constant shaking, till the bromine colour remains ; allow to stand for half an hour ; if the colour disappears run in more bromine water from the burette ; shake again and allow to stand ;

note the total amount added; then add excess of a solution of potassium iodide; and determine the liberated iodine with a standard solution of thiosulphate in the usual way, as described on p. 6. The free iodine is equivalent to the excess of bromine and must be subtracted from the total bromine which was added. The amount of bromine water necessary to react with the 0·25 gram of pure phenol is thereby obtained, and from this number the phenol-equivalent for every cubic centimetre of the bromine solution is calculated. The equation representing the chemical change is

$$C_6H_5OH + 3Br_2 = C_6H_2Br_3 . OH + 3HBr.$$

The experiment is then repeated with the sample of phenol which is being analysed, and thus obtain the strength of the phenol.

The Lancet Acetone-baryta (L.A.B.) method of the chemical analysis of phenol disinfectants.

The majority of the liquid coal tar disinfectants are mixtures of varying quantities of phenolic bodies with inert tar oils, in many cases with soap or resins or other emulsifying agents such as dextrin or gelatin, etc. In connection with The Standardization of Disinfectants, a very full account is given in the *Lancet* for Nov. 13, 20, and 27, 1909, by Professor Sims Woodhead, Dr C. R. Ponder, Mr S. A. Vasey and Mr J. E. Purvis, the members of the Commission appointed by the *Lancet* to investigate the conditions of standardization. The inquiry consisted of two parts, (*a*) the chemical analysis of disinfectants and (*b*) the bacteriological examination of disinfectants. The methods of the chemical examination only are described here.

(*a*) Fluids containing soaps and resins as emulsifiers. The basis of the method is that the soaps and resins must be decomposed or made insoluble before there can be a complete separation of the phenoloid bodies in a pure state. This may be done by converting the soaps or resins, which are commonly of soda or potash, into insoluble combinations by the addition of a strong solution of barium hydroxide. Also, phenol, cresol, and the phenolic bodies generally are soluble in this alkaline solution. Lime water may be used, but calcium hydroxide is considerably less soluble in water than barium hydroxide.

If a few grams of the disinfectant, which is made by amalgamating phenol bodies with soaps or resin (the constitution of most of the disinfectants sold), be first hydrolysed by stirring with water and then an excess of baryta water added, the actual germicidal body is completely dissolved in the baryta, and there is a rapid separation of the resins or soaps in the form of insoluble baryta compounds.

The method is to weigh out 10 grams of the disinfecting fluid (composed of phenol bodies, soaps or resins, and neutral oils); shake well with 100 grams of distilled water; add 15 grams of barium hydrate crystals; place the mixture in a large conical flask attached to a reflux condenser; heat for half an hour at 100° C. by immersing it in a vessel of boiling water; shake the flask at frequent intervals; allow to cool. In most cases the baryta solution can be decanted almost clean without filtering; but it is best to pour it upon a moistened asbestos plug in a glass funnel which is attached to a water-pump. The residue in the flask is then washed with warm baryta solution, and the whole filtered. Make up the filtrate to 300 c.c. Take 50 c.c. of this one treated in a separating funnel with hydrochloric acid, calcium chloride added, and the liberated phenols extracted and washed with ether, and evaporate off the ether. The residue which consists of the phenol bodies is weighed. Then dissolve in sodium hydroxide and add excess of a solution of bromine in caustic soda, the amount of bromine present in the solution being ascertained previously by Koppeschaar's process (p. 180); the bromine is liberated by adding hydrochloric acid; the bromine absorbed by the phenol is thereby found. Care should be taken that the bromine is in excess, and the excess is determined by influxing it with iodine by the addition of a few crystals of potassium iodide, and estimating the iodine by N/10 thiosulphate of soda in the usual way. The amount of bromine which has reacted with the phenol body is thus determined and calculated in terms of its equivalent of carbolic acid according to the equation:

$$C_6H_5OH+3Br_2=3HBr+C_6H_2Br_3OH.$$

If the phenol body present is carbolic acid the percentage

figure obtained in the bromine experiment will approximate to the percentage weight of carbolic acid found by drying the ether extraction of the acidified baryta solutions. If there is no agreement between these figures carbolic acid as such is not present but one or other of its homologues or a phenoloid, the bromine absorption of these bodies being lower. When, in fact, the difference is wide, the conclusion may be justified that carbolic acid is absent. The residues after treatment with baryta, which are generally of a viscous description when soap is present but hard and firm when resins are present, contain the neutral hydrocarbon oils, the fatty acids and the resins. To this residue add 200 c.c. of acetone; separation gradually follows, and the neutral hydrocarbon oils dissolve in the acetone, the soaps and resins are insoluble. Thoroughly rub down the insoluble matters with acetone, and filter and wash with acetone; treat the insoluble matter with hydrochloric acid and shake up with ether; evaporate the ether, and weigh the fatty acids or resins thus obtained. To the acetone solution add 10 per cent. solution of caustic soda and mix well and then about 20 c.c. of petroleum (white spirit) added to facilitate the removal of oils, and finally add about 500 c.c. of water; shake and allow to stand; draw off the aqueous liquid and filter through a wetted filter paper; add hydrochloric to the filtrate till it is acid, and exhaust with ether, the residue is the resins or fatty acids which were not retained in the baryta residue. The amount is usually small and should be added to the weight of the fatty acids or resins found in the barium residue.

Any water present may be determined by taking 25 grams of the disinfectant fluid, and add exactly 10 c.c. of a 10 per cent. solution of sulphuric acid and then 25 c.c. of petroleum spirit; shake thoroughly and allow to rest. When the separation is complete, the clear liquid below is drawn off with a narrow and accurately graduated glass cylinder and its volume read; the excess over the 10 c.c. of aqueous sulphuric acid added is regarded as the water present in 25 grams of the sample.

The alkalis are estimated by obtaining the ash and titrating the solution of this residue with standard acid.

(*b*) Fluids containing neither soaps nor resins as emulsifiers.

The germicidal agent of these disinfectant fluids is brought into uniform suspension by means of gelatin or gum. In such cases the use of baryta is unnecessary. The method is to take 10 grams of the fluid and add excess of absolute alcohol or acetone which dissolves the phenoloid, but precipitates the gelatin or dextrin ; thoroughly exhaust the insoluble mass with successive washings of acetone and finally dissolve in water ; evaporate the solution to dryness, and then weigh the residue of gelatin or gum so obtained. The alcohol or acetone containing the phenoloids (and neutral oils if present) is then shaken up with a 10 per cent. solution of sodium hydroxide and diluted freely with water. Any neutral oils present are thus set free, and this may be promoted by adding 20 c.c. of petroleum (white spirit). The alkaline solution is then drawn off, filtered, and the filtrate made up to a known volume. Part of this is acidified and calcium chloride added ; exhaust the separated phenoloids with ether ; evaporate off the ether ; and the phenoloid weighed. After weighing the phenoloid, dissolve in sodium hydroxide and examine for its bromine value as described above.

The water in these preparations is determined in the same way as in the analysis of disinfectants containing soaps and resins, but a longer time is required for the complete separation of the water from the phenoloids and oils owing to the difficulty of breaking up the emulsions.

CHAPTER XII

AIR—COAL GAS—OTHER GASES

AIR.

Air consists of a mixture of oxygen, nitrogen, carbon dioxide and water vapour, together with small quantities of the inert gases, argon, krypton, neon, xenon, etc. There are present in the air about 20 per cent. of oxygen, and 80 per cent. of nitrogen. Air has been examined from the tops of mountains, from the middle of large cities, from the depths of valleys and from the open prairie and from many other places, but the proportions of oxygen and nitrogen remain practically the same in all places. The carbon dioxide, on the other hand, varies considerably, and is influenced by the local conditions; respiration of animals removes oxygen and adds carbon dioxide, expired air containing about 5 per cent. of carbon dioxide; coal, gas, wood, etc. when burned use up the oxygen and add carbon dioxide. One litre of pure air at 0° C. and 760 mm. pressure weighs 1·29366 grams, and the air nearer the surface of the earth is more dense than that further away since it is subjected to greater pressure.

Ozone. There are usually only traces of ozone present in air and reliable methods for its determination are not known. A qualitative test for the presence of ozone has been proposed by Engler and Wild (*Ber.* 1896, XXIX, 1940), in which a large volume of the sample is passed first over chromic acid to remove the hydrogen peroxide, and then over manganese sulphate paper and thallous oxide paper; if both papers are coloured brown, ozone is present.

Test for ammonia. Ammonia is present in the atmosphere in traces from the putrefaction of organic matter containing nitrogen. For a qualitative test, large quantities of the sample are passed over moist litmus or turmeric paper, when, in the

presence of ammonia, the litmus paper becomes blue, and the turmeric paper brown. A rough estimation of the amount of ammonia in the air may be obtained by passing a large volume of the sample through distilled water and estimating the ammonia, absorbed by the water, with Nessler's reagent as described under water analysis (p. 3).

Estimation of organic matter. A known volume of the sample is passed through a tube in which is placed a plug of clean glass wool, which is then stirred into 100 c.c. of potassium permanganate solution (containing 0·395 gram per 100 c.c.), and 100 c.c. of dilute sulphuric acid (1 in 3); the mixture is then digested for 1 hour at 60° F., 10 c.c. of the potassium permanganate solution are then titrated with the standard oxalic acid solution (p. 6); a blank experiment is at the same time performed, and the number of c.c. of the standard oxalic acid solution used in the actual estimation deducted from the number of c.c. used for the blank gives the number of c.c. of potassium permanganate solution used in the estimation. Each c.c. of potassium permanganate solution used = 0·0001 gram of oxygen absorbed by the organic matter in the sample. The results are expressed in terms of oxygen absorbed and are only comparative.

Estimation of dust. A large volume of air is drawn through a tube containing a weighed piece of cotton wool; the increase in weight of the wool represents dust.

Nitrogen. Nitrogen is always determined by difference and is left as a residue after the removal of all the other constituents.

Estimation of oxygen. *Solution required:* An alkaline solution of pyrogallol containing 5 grams of pyrogallol and 120 grams of potassium hydrate in 100 c.c. of water.

The process. The determination is made in Bunte's burette, which consists of a graduated cylinder closed by a tap at both ends; the tap at one end has a double bore, one of which connects with an inlet tube and the other with a glass cup; the burette is placed in a vessel of mercury with both taps open and is thus filled with mercury; the holder containing the sample is connected by a piece of rubber tubing to the inlet tube and the burette is raised partially out of the trough;

the mercury in the burette sinks and draws the air from the holder into the burette; the tap at the inlet tube is now closed and the holder is disconnected; the volume of the gas in the burette is ascertained by raising or lowering the burette in the mercury until the levels of the mercury inside and outside the burette are the same; the volume is then read off on the graduations, and the temperature of the surrounding air is noted and also the height of the barometer. The alkaline pyrogallol solution is now placed in the cup, which is connected with the burette, and the solution allowed to run in, care being taken that no air enters with it; both taps are now closed and the burette is removed from the mercury trough and shaken to mix thoroughly the solution and the air; the burette is replaced in the mercury and the bottom tap opened, when the mercury will rise in the burette to take the place of the absorbed oxygen; the tap is again closed, and the contents of the burette shaken until the mercury ceases to rise on opening the tap; the levels of the mercury inside and outside the burette are then adjusted, and the volume of the residual gas noted and also the temperature and the barometric pressure; the volume is then reduced to 0° C. and 760 mm. pressure; the decrease in volume represents oxygen.

Carbon dioxide. Since carbon dioxide is one of the products of respiration it will accumulate in the air of closed and occupied rooms. It is necessary, therefore, to have a simple method of determining the proportion of carbon dioxide in air in order that a judgment may be formed of the efficiency of the ventilation. This is done by the well-known Pettenkofer's process.

Solutions required: Saturated lime water solution. Standard oxalic acid solution containing 2·8636 grams of pure dry crystallized oxalic acid per 1000 c.c. Each c.c. of this solution represents 0·001 gram of oxygen.

The process. A glass bottle, holding about 8 to 10 litres and closed with a tight-fitting rubber bung, is obtained and the exact volume of the bottle is determined by filling it with water and measuring the volume of the water contained. The bottle, which must be perfectly clean and dry, is then filled with the air to be tested; 100 c.c. of the lime water are run

into the bottle and the bung at once inserted and the temperature and pressure noted ; the lime water is then mixed with the air in the bottle by shaking, and this is repeated occasionally during one hour ; the lime water is then rapidly poured into a stoppered 200 c.c. flask ; the bottle is rinsed once or twice with distilled water, free from carbon dioxide ; the washings are added to the flask and the whole is made up to 200 c.c. with distilled water, free from carbon dioxide ; 50 c.c. of this solution are then titrated with the standard oxalic acid solution, using phenol phthalein as an indicator, until the pink colour is just discharged ; the number of c.c. of the standard oxalic acid solution used multiplied by four gives the number of c.c. required for the 100 c.c. of lime water taken. 50 c.c. of the original lime water are now similarly titrated with the standard oxalic acid solution and the number of c.c. used multiplied by two gives the number of c.c. required for 100 c.c. of the solution. The difference in the volume of acid used before and after the experiment is thus obtained ; and this difference multiplied by 0·001 gives the weight of carbon dioxide in the volume of air taken. Care must be taken that throughout the experiment the lime water does not absorb any carbon dioxide from the breath or the surrounding atmosphere.

Example : The capacity of the bottle was 10,300 c.c. ; the temperature of the sample was 20° C. and the pressure 750 mm. ; 100 c.c. of the original lime water required 95·8 c.c. of the standard oxalic acid solution and after the experiment 88·7 c.c., then $95{\cdot}8 - 88{\cdot}7 = 7{\cdot}1 \times 0{\cdot}001 = 0{\cdot}0071$ gram of carbon dioxide. 44 grams of carbon dioxide occupy 22,400 c.c. at 0° C. and 760 mm., therefore the volume of the carbon dioxide $= \frac{0{\cdot}0071 \times 22{,}400}{44} = 3{\cdot}61$ c.c. at 0° C. and 760 mm., therefore the volume at 20° C. and 750 mm. $= \frac{3{\cdot}61 \times 293 \times 750}{273 \times 760} = 3{\cdot}82$ c.c., then $\frac{3{\cdot}82 \times 100}{10{,}300} = 0{\cdot}37$ per cent. of carbon dioxide in the sample.

Haldane's method. A small and compact instrument has been devised by Haldane to determine the amount of carbon

dioxide in air. It requires a little practice to get accurate results. The method consists in absorbing the carbon dioxide from 25 c.c. of air by caustic potash, and then measuring the decrease in volume under the same conditions of temperature and pressure. Each of the graduated parts of the burette is divided into 1/10,000 part of the whole capacity of the burette, so that the results are read off in parts per 10,000. The chemical change is, of course,

$$2KOH+CO_2=K_2CO_3+H_2O.$$

It is obvious that great care must be taken in the experiment with such small changes in volumes.

Lunge and Zeckendorf's method (*Zeitsch. Angew. Chem.* 1888, 1, 395). This is an excellent rapid method for the approximate determination of carbon dioxide, especially when it is present in large quantities.

Solution required: N/10 sodium carbonate solution containing 5·3 grams of sodium carbonate, and 0·1 gram of phenol phthalein in 1000 c.c.

Dilute sodium carbonate solution; 2 c.c. of the above solution are placed in a 100 c.c. graduated flask, and made up to the mark with distilled water, free from carbon dioxide, and the flask instantly closed; this dilute solution will not keep for more than 12 hours.

The process. A wide-mouthed bottle, of 110 c.c. capacity, is fitted with a stopper in which are two holes through which pass two glass tubes, one reaching to the bottom of the bottle and attached to a rubber bulb of 70 c.c. capacity; fill the bottle with some of the air to be examined by compressing the rubber bulb several times; then place in the bottle 10 c.c. of the dilute sodium carbonate solution and immediately replace the stopper; the contents of the rubber bulb are now driven through the solution in the bottle, and the contents of the bottle are shaken; this is continued until the pink colour has been discharged; the amount of carbon dioxide is then obtained from the following table by noting the number of compressions of the bulb required to discharge the pink colour.

Compressions	CO_2 in parts per 1000	Compressions	CO_2 in parts per 1000	Compressions	CO_2 in parts per 1000
2	3·00	11	0·87	20	0·62
3	2·50	12	0·83	22	0·58
4	2·10	13	0·80	24	0·54
5	1·80	14	0·77	26	0·51
6	1·55	15	0·74	28	0·49
7	1·35	16	0·71	30	0·48
8	1·15	17	0·69	35	0·42
9	1·00	18	0·66	40	0·38
10	0·90	19	0·64	48	0·30

Aqueous vapour. The dew-point, or the temperature at which the aqueous vapour in the air begins to be deposited, is determined by Daniel's hygrometer or by a wet-and-dry bulb thermometer. In the latter instrument the temperature of the dry bulb is noted, together with the temperature of the other bulb upon which the film of moisture was first seen to be deposited. Certain factors have been worked out by Glaisher, and by means of his tables it is easy to calculate from the temperature observations the amount of aqueous vapour by weight in a definite volume of air. These details are given in most books dealing with the general principles of hygiene.

Ground air. There is a considerable amount of carbon dioxide in ground air with small quantities of ammonia and also various hydrocarbons, and sulphuretted hydrogen. The gases may be collected by Hesse's method. A narrow steel cylinder with a sharp point, and perforated with numerous holes just above the point, is forced into the earth to varying depths as required. The outside end of the tube is connected with a large glass vessel of 8 to 10 litres capacity, and this is connected in turn with an aspirator. The jar is first emptied of its air by means of the aspirator and the connection between the steel tube and the glass vessel is re-established by opening the stop-cocks. The ground air is then aspirated into the glass vessel and the sample analysed for carbon dioxide as described above. Another series of experiments by aspirating the ground air through pure water will dissolve the ammonia and this can be determined by Nessler's solution in the usual way. The organic matter in the ground air can also be determined similarly by passing the gases through a known strength of a dilute solution of potassium permanganate, as described on p. 186.

Carbon monoxide. Vogel (*Ber. Chem. Gesellsch.* XI, 235) was the first to use the well-known spectrum of blood charged with carbon monoxide as a means for detecting the gas. The method is to take 2 or 3 c.c. of blood, dilute it with water till only a very faint red colour is seen; place in a dry bottle and aspirate about 10 litres of the suspected air through it. When carbon monoxide is present the blood at once becomes rose coloured, and upon the addition of two drops of a freshly prepared and colourless solution of ammonium sulphide and shaken the well-known absorption bands do not disappear; in blood which is free from carbon monoxide the absorption bands are replaced by a broad and weakly defined band. The ammonium sulphide should always be prepared as it is wanted, by passing sulphuretted hydrogen through a solution of ammonia. The comparison of the two spectra is best effected by placing the two solutions in test-tubes, and using the test-tubes as condensing lenses. One is placed in front of the slit end of the spectroscope, and the other in front of a small reflecting prism which covers the slit about half its length. Two small fish-tail burners are used as the sources of light, and these are placed end on and not with their broad fronts facing the slit; then, by carefully adjusting the two test-tubes containing the fluids, two sharp images can be easily obtained, and the spectra examined. The slit should not be too wide, otherwise the light is scattered and overlaps the absorption bands and their sharpness is lessened.

Hartridge (*Jour. of Physiology*, vol. XLIV, p. 1) describes a spectroscopic method for the estimation of CO. One of the advantages claimed is that two collimators are used instead of one. "These are placed symmetrically one on either side to the normal to a grating surface in such a position that the first order of the spectrum from each occupies the centre of the visual field. But it is found with this arrangement that the spectra are reversed in direction so that the red of one lies over the violet of the other, the blue over the orange and the yellow over the green. If however the one collimator be placed at a slightly higher level than the other, then these spectra, instead of overlying may be so placed that their margins just touch.

Having obtained this arrangement it is possible by moving the collimators to place any one wave length in one spectrum in a line with a similar wave length in the other, so that for instance the D lines or any other lines might be made to correspond. From a knowledge of the grating constant and the angle between the collimators, the wave length of the line or band in adjustment may at once be determined. Two instruments have been employed, both of which are modifications of a direct vision spectroscope. The first had two slits and consisted essentially of a small square box at one end of which these were mounted; at the other was the grating by which the spectra were formed. Outside the box and immediately in front of the slits are the vessels containing the blood samples. In front of the grating and between it and the eye is a lens which helps to focus correctly the spectra. The position of this lens is contrary to spectroscopic practice, but was done to avoid the astigmatism this lens would cause if placed between the grating and the slits. The second instrument had but one slit, the second spectrum being obtained by the employment of a reflecting prism; the light paths were so arranged that they should still be approximately equal. The advantages gained by this arrangement are obvious, the use of but one light and one blood sample. In both instruments the slit or slits are permanently fixed in place, a micrometer being used for changing the wave length."

As the method depends upon the visual judgment of the observer, and is therefore affected by the brightness of the source of light and the state of adaptation of the eye, the author suggests various precautions which should be observed, such as the use of a ruby light to illuminate the room. He also used various thicknesses of the solution by employing blood tubes made from glass of oval section, so that by using these at the long or short axis, one could alter the amount of blood the light had to traverse in reaching the slit.

Haldane's method of detecting and estimating carbon monoxide in air (*Jour. of Physiology*, vol. XVIII, p. 461). The method depends upon the fact that when a hæmoglobin solution is well shaken with air containing CO, the proportion of the

hæmoglobin, which finally combines with the CO, varies with the percentage of CO present in the air. By estimating colourimetrically the proportion of the hæmoglobin which has combined with the CO it is thereby possible to infer the percentage of CO present in the air. In employing hæmoglobin for the purpose Haldane proceeds as follows: "The sample is collected by sucking two or three litres of the air through a clean and dry bottle of about 100 to 200 c.c. capacity. The bottle is closed by means of a doubly tubulated cork, previously soaked in paraffin. Each of the pieces of glass tubing which pass through the cork is securely closed by means of a stopper made of about an inch of rubber tubing, into one end of which fits a piece of glass rod. For the determination of the CO about 5 c.c. of a solution of defibrinated ox blood diluted to 1/100th with water, are introduced into the bottle in the following manner. A pipette having been filled with the blood solution one of the pieces of glass rod is removed, the rubber tubing being at the same time compressed, so as to prevent air from passing into the bottle. The end of the pipette is now inserted into the rubber tubing, and the blood solution allowed to flow into the bottle. As the increase of pressure within the bottle prevents more than a part of the blood solution from entering, it is necessary to allow some of the air inside to escape. This is easily effected by pinching the rubber tubing which fits in the other glass stopper, and so opening a small channel for the passage outwards of the air. The pipette having been removed and the glass stopper again inserted without letting any air in the bottle is now gently shaken for about 10 minutes, at the end of which time the hæmoglobin will have taken up all the CO which it is capable of taking up from the contained air. With vigorous shaking the time required is much less, but the solution may become turbid from mechanical coagulation of albumin and hæmoglobin, so that gentle shaking for a longer time is to be preferred. When the shaking is complete the solution is removed with a pipette into one of three narrow test-tubes of exactly equal diameter (about 3/8 or 1/2 inch), and each capable of holding at least 12 c.c. Into another of the test-tubes exactly 5 c.c. of the original blood solution are

measured by means of a pipette. The third test-tube is filled with some of the same blood solution, the hæmoglobin of which has been already saturated with CO. The saturation is effected by placing some of the blood solution in a small bottle or flask, the air in which is then washed out with coal gas. When the bottle is full of coal gas it is quickly closed with the thumb, and then shaken for 2 or 3 minutes so as to saturate the hæmoglobin with CO, of which coal gas contains about 5 %—a quantity sufficient to produce (particularly in the absence of oxygen) rapid and absolutely complete saturation. The test-tube containing the saturated blood should be filled full and corked if it is to be kept any time, as otherwise the CO escapes from the solution. The tints of the solutions in the test-tubes are now compared. If more than 0·01 % of CO was present in the sample of air the blood solution which has been shaken with the air will be pinker than the original solution contained in the second tube. If more than 3 % of CO was present the tint will be sensibly equal to that of the solution saturated with coal gas. An approximate estimate of the saturation may even be made by simply comparing the three tubes, and from this estimate the percentage of CO in the sample of air may be roughly estimated from the table on p. 196.

"Standard carmine solution is now added from a burette to the solution in the second test-tube, until its tint is exactly equal to that of the solution which was shaken with the air. When the tints are nearly equal not more than 0·25 c.c. of carmine solution are added at a time. The points are noted at which there is just not enough, and just too much carmine. the mean being taken as the correct result. The quantity of carmine required to produce equality of tint with the saturated solution is also determined or redetermined at the same time. The carmine solution is prepared as follows. One gramme of pure carmine is mixed in a mortar with a few drops of ammonia and dissolved in 100 c.c. of glycerine. This solution is preserved and a standard solution prepared from it when required by diluting 5 c.c. of the glycerine solution to 500 c.c. with water. It will be found that when about 5 c.c. of this standard carmine

solution are added to 5 c.c. of ox blood diluted to 1/100th, the tint of the resultant solution is the same both in quality and intensity as that of the diluted blood when saturated with CO or coal gas. Before making a determination, however, it is well to ascertain that the two solutions actually correspond. If in order to produce equality in intensity as well as quality of tint it is necessary to add water to either test-tube, the stock of blood solution or carmine solution must be diluted accordingly. It will be found that the amount of standard carmine solution required to produce the saturation tint varies slightly as the tint of the daylight changes. For this reason the amount should be determined each time the solution is used. The standard carmine solution should be prepared fresh when required, as it distinctly loses strength when kept for a few days. The blood solution must also be freshly prepared, although the blood itself may be kept for a long time in a corked bottle. A blood solution may be so far altered as to be unfit for use even when the oxyhæmoglobin bands, and no others, are visible on spectroscopic examination. A solution thus altered does not change colour to the usual extent when saturated with CO.

"From the results of the titration with carmine solution the percentage saturation of the hæmoglobin with CO may be easily calculated in the manner illustrated by the following example. To 5 c.c. of blood solution 6·2 c.c. of standard carmine were required to be added to produce the tint of blood solution shaken with the air under examination. In the former case the carmine was in the proportion of 6·2 to 11·2; in the latter case in the proportion of 2·2 to 7·2. The percentage saturation of the hæmoglobin in the blood shaken with the air was thus

$$\frac{2{\cdot}2}{7{\cdot}2} \times \frac{11{\cdot}2}{6{\cdot}2} \times 100 = 55{\cdot}2.$$

"The method will only give correct results when the oxygen percentage in the air is nearly normal. A deficit of two or three per cent. of oxygen will hardly influence the result appreciably, and may therefore be disregarded. A deficit of 5 per cent. in the oxygen of the air will, however, cause the results

to be almost a third too high, so that, for example, half saturation of the blood solution would apparently indicate 0·15 % when in reality only 0·11 % was present."

Also, when there is more than 0·5 CO per cent. present in the air, or when there is little or no oxygen present, the sample must be suitably diluted with air. Coal gas, for example, would require to be diluted 1/40th or 1/50th with air.

In a later paper (*Jour. of Physiology*, vol. XX, p. 521), Haldane states that daylight has a marked influence on the stability of carboxyhæmoglobin, and therefore recommends that the blood solution and sample of air must be covered with a cloth during the process of shaking, and a very bright light should be avoided during the process of titration with the carmine solution. The following table gives as nearly as possible the values when the bottle is protected from light during the shaking, and from this table the percentage of CO in the sample of air may be calculated.

Percentage saturation of hæmoglobin in blood solution	Percentage of CO in the air	Percentage saturation of hæmoglobin in blood solution	Percentage of CO in the air
5	0·006	55	0·11
10	·012	60	·135
15	·019	65	·160
20	·026	70	·21
25	·034	75	·27
30	·043	80	·36
35	·054	85	·51
40	·066	90	·81
45	·078	95	1·70
50	·090		

COAL GAS.

The analysis of coal gas may be accomplished by means of Hempel's gas apparatus. It consists of a burette, which includes a measuring tube and a pressure tube, and a pipette used as an absorbing vessel for the various constituents. The gaseous mixture is subjected to the action of suitable absorbents in turn in a series of the pipettes, whereby the proportions of the constituent gases in the mixture can be estimated.

The burette is filled with the gas and connected with the pipette. The stop-cock between the burette and the pipette is opened; the pressure tube connected with the former is

raised, and the gas flows into the pipette; turn off the stop-cock when the water in the burette has reached the required mark in the capillary tube connecting the burette with the pipette; shake the pipette gently for a minute or two and repeat the operation; transfer the gas back to the burette by opening the stop-cock, and read off the volume of gas after bringing the height of the water in the pressure tube to the same height as that in the burette. Subsequent absorptions are repeated similarly, each constituent gas being absorbed by a special reagent. A separate pipette is kept for each reagent. In coal gas the constituent gases are absorbed in the following order:

(1) Carbon dioxide, by caustic potash solution.

(2) Olefines, by saturated bromine water; and then remove the bromine vapour by caustic potash solution in a separate pipette.

(3) Benzene, by fuming nitric acid; and then remove the oxides of nitrogen by the caustic potash pipette.

(4) Oxygen, by a solution of alkaline pyrogallic acid.

(5) Carbon monoxide, by a freshly made solution of ammoniacal cuprous chloride.

(6) The residual gas is transferred to a pipette charged with water, and the hydrogen and methane are estimated by mixing with oxygen and sparking in a special form of Hempel explosion pipette. The gases are thus burnt to carbon dioxide and water.

(7) Nitrogen and the argon series of gases are estimated as a residue.

Estimation of ammonia and total sulphur. The gas is tested qualitatively for ammonia and sulphuretted hydrogen by passing it over moist lead acetate paper, and moist red litmus paper; in the presence of sulphuretted hydrogen the lead acetate paper becomes dark brown or black, and, in the presence of ammonia, the red litmus paper becomes blue.

Quantitative determination. The gas is first passed through a cylinder, containing glass beads, which have been moistened with a known quantity of N/10 sulphuric acid; during the passage through this cylinder the ammonia combines with the

sulphuric acid to form ammonium sulphate. The gas is then passed through a burner and burned at the rate of 0·5 cubic feet per hour until 10 cubic feet have been burned ; the burner is surrounded by pieces of solid ammonium carbonate and the gases formed by the combustion are passed through a trumpet tube into a cylinder filled with glass balls which are kept moist ; the cylinder is connected with a long glass chimney in which the final condensation takes place ; the sulphur dioxide formed by the combustion of the sulphur in the gas dissolves in the water and passes to the bottom of the cylinder, and through the bottom of the cylinder into a glass beaker ; after the combustion is completed the cylinder is washed out with distilled water and the washings are heated with a few drops of bromine to complete the conversion of all the sulphur into sulphate ; the liquid is then made acid with hydrochloric acid and an excess of barium chloride solution is added ; the precipitated barium sulphate is collected on a filter paper, washed with distilled water, dried in a water oven, incinerated and weighed. The weight of barium sulphate obtained multiplied by 0·1373 gives the amount in grams of total sulphur in 10 cubic feet of the gas. The sulphur is usually returned as grains of sulphur per 100 cubic feet of gas.

Estimation of ammonia. The first cylinder, containing the N/10 sulphuric acid, is washed out with distilled water, and the excess of N/10 sulphuric acid is determined by titration with N/10 sodium hydroxide solution. The number of c.c. of N/10 sulphuric acid neutralized during the passage of the gas multiplied by 0·0017 gives the number of grams of ammonia in 10 cubic feet of the gas.

Estimation of sulphuretted hydrogen. Somerville (*Jour. Gas. Light.* 1912, CXII, 29). *Solutions required:* N/1000 iodine solution. Starch solution.

The process. 10 c.c. of the N/1000 iodine solution and 10 c.c. of the starch solution are placed in a 100 c.c. wash bottle, and the gas is passed through the mixture by means of an aspirator until the blue colour disappears ; the volume of the gas used is determined by measuring the volume of the water run out of the aspirator. A second determination is then made

in which the gas is first passed through a scrubbing tower, containing lead carbonate, which removes all the sulphuretted hydrogen; the apparent sulphuretted hydrogen obtained in the second experiment is subtracted from the amount obtained in the first experiment, as this apparent sulphuretted hydrogen is due to the unsaturated compounds, hydrogen cyanide, etc., present in the gas, which combine with the iodine. 10 c.c. of the N/10 iodine solution represent 0·01122 c.c. of sulphuretted hydrogen.

Determination of illuminating power. *Apparatus required:*

(1) Standard light, which is a 10 candle power pentane lamp.

(2) Standard burner, which is a Number 2 Argand burner.

(3) Standard photometer, which is a bar photometer, or a jet photometer.

The process. The pentane lamp and the standard burner are fixed at opposite ends of the photometer in such a manner that the tops of the two flames are equidistant from the table; the pentane lamp is lighted and allowed to burn for 15 minutes; the argand burner is then lighted, and the gas consumption regulated to 5 cubic feet per hour; a box containing a piece of paper on which is a grease spot between the two lights is then moved about on the bar until the grease spot is equally illuminated on both sides, which is shown by the grease spot becoming invisible; the illuminating power is then read off on the bar in candle power.

Calorific value of coal gas. Coal gas is now largely used for heating and cooking purposes, and its calorific value is usually determined by the Public Analyst. The calorific value is the amount of heat given out by the complete combustion of a known volume or mass of the gas. The unit employed is known as the *calorie*, and it is defined as the amount of heat necessary to raise 1 kilogram of water from 0° to 1° C. Another unit is called the *British Thermal Unit* and it is defined as the amount of heat required to raise 1 lb. of water 1° F. One calorie is equal to 3·9683 British Thermal Units.

The calorific value is determined experimentally by burning

a definite volume of the gas under conditions whereby measurements can be made as to the amount of heat given off when it is burnt. The gas is conducted through an experimental meter and it is burnt at rates varying from 3 to 30 cubic feet per hour. Coal gas is usually burnt at about 6 cubic feet per hour. The burner is placed within the cavity of a double-walled cylinder, and the burnt gases escape through tubes which pass through the space between the walls of the cylinder. The space round the cylinder contains water which flows in at the bottom and out at the top, so that it is in contact with the tubes down which the burnt gases descend. Delicate thermometers indicate the temperatures of the incoming and outgoing water. The rate at which the water is supplied is regulated so that the exit water is from 10° to 20° warmer than the water which enters. A large measuring cylinder of 2 litres capacity is used to collect the water. The temperature of the outflowing water is observed till it remains constant, and the long hand of the experimental meter is observed till it is just passing the zero mark, when the water is collected in the 2-litre measuring flask. The temperature of the outflowing water is observed six times while the glass is filling, and the temperature of the inflowing water about twice. After 2 litres of the water have been collected, the gas supply is cut off and the meter is read. This gives, therefore, the amount of gas which has been burnt during the filling of the flask. The average temperature of the water which goes in and that which comes out is also known; and therefore we can estimate by how many degrees the combustion of a definite volume of the gas has raised the two litres of water. To find the calorific value of unit volume of the gas, twice the mean rise in temperature of the water is divided by the volume of the gas which has been burnt. As the gas is usually measured in cubic feet, the result is the calorific value of a cubic foot of gas.

The water condensed from the combustion of the gases collects at the bottom of the apparatus, and it is run off and measured in a small measuring cylinder. For every c.c. of water so condensed per cubic foot of gas burnt, a deduction of 0·6 calorie is made from the calorific value previously found.

This is to eliminate the value of the heat given off on the condensation of the steam to water; because the water produced by the combustion of gas escapes as steam with the other products of combustion, and therefore its latent heat is not available for use.

The following analyses of an illuminating gas have been kindly supplied by Mr West Knights.

	(1)	(2)	(3)	(4)
Hydro-carbons (ethylene, etc.)	4·0	4·6	3·5	3·8
Oxygen	·5	·2	·5	·7
Carbon monoxide ..	5·7	6·2	5·6	5·6
Carbon dioxide	nil	nil	nil	nil
Methane	33·9	30·8	34·0	32·6
Hydrogen	51·8	56·1	51·6	52·7
Nitrogen	4·1	2·1	4·8	4·6
	100·0	100·0	100·0	100·0

Sulphur, in grains per 100 cubic feet.

Aug. 1911	17·32 grains	The sulphur in these three instances is very high.
Jan. 1912	24·10 ,,	
,, 1913	10·40 ,,	
,, 1914	7·95 ,,	

There was no sulphuretted hydrogen present.

Illuminating power.

Aug. 1912	17·22 candles
Jan. 1913	16·82 ,,
Sept. 1913	17·08 ,,
Jan. 1914	16·33 ,,

The legal standard is 16 candles.

CHAPTER XIII

RAG FLOCK—URINE

EXAMINATION OF RAG FLOCK.

The Rag Flock Act, 1911, provides that "it shall not be lawful for any person to sell or have in his possession for sale flock, manufactured from rags, or to use for the purposes of making any article of upholstery, cushions or bedding, flock manufactured from rags, or to have in his possession flock manufactured from rags intended to be used for any such purpose

unless the flock conforms to such standard of cleanliness as may be prescribed by the regulations to be made by the Local Government Board." The Rag Flock Regulations, 1912, were issued by the Local Government Board under the above Act and came into force on July 1st, 1912, they provide that " Flock shall be deemed to conform to the standard of cleanliness for the purposes of the Rag Flock Act, when the amount of soluble chlorine in the form of chlorides, removed by thorough washing with distilled water at a temperature not exceeding 25° C. from not less than 40 grams of a well mixed sample, does not exceed 30 parts of chlorine per 100,000 parts of flock." Warburton has shown (*Reports to the Local Government Board on Public Health and Medical Subjects*, No. 27) that " lice and their eggs may pass through the flocking machine without being crushed. The lice themselves are incapable of surviving more than 3 or 4 days without food, and it is extremely unlikely that any of them would be alive when the flock were converted into bedding. Their eggs however may take a month or more to hatch and it is quite possible that living eggs might be present in the bedding."

Chemical examination of rag flock. Garrett (*Reports to the Local Government Board on Public Health and Medical Subjects*, No. 27), adopts the following process: The flock is spread out to dry at the temperature of the room; and, after 24 hours, the moisture is determined by drying at 105° C. for 90 minutes. 100 grams of the sample are then placed in a large beaker, and 3000 c.c. of water are added; the mixture is allowed to stand for 39 minutes with frequent stirring; the flock is then strained and wrung out; the liquid is filtered and the filtrate examined as if it were a sample of water; the total solid matter, chlorine and free and albuminoid ammonia are estimated.

Estimation of soluble chlorine. Parkes (*Chem. News*, 1913, CVIII, 177). *Solutions required:* Silver nitrate solution. Weigh out 4·8 grams of silver nitrate, dissolve it in water, and make the solution up to 1000 c.c. Each c.c. of this solution represents 0·001 gram of chlorine.

The process. 50 grams of the well mixed sample are placed in a large beaker and 500 c.c. of water are added; the flock

is allowed to steep for 4 hours and is then drained ; pressed by hand and washed with successive quantities of water (of 100 c.c. each) until 1000 c.c. of washings are obtained ; the washings are then filtered and 100 c.c. of the filtrate (= 5 grams of flock) are evaporated to dryness in a porcelain dish with a minute quantity of lime ; the residue is then charred, cooled, extracted with water and filtered ; a few drops of potassium chromate solution are added to the filtrate, and the standard silver nitrate solution drop by drop from a burette until a permanent red colour is obtained. Each c.c. of silver nitrate solution used multiplied by 20 gives the parts of chlorine per 100,000 parts of flock. In the case of unwashed flock it is advisable to add a few drops of phenol solution as a vermicide.

Example : Treated as described above the filtrate from a sample of rag flock required 17·3 c.c. of the standard silver nitrate solution ; then 17·3 × 20 = 346 parts of soluble chlorine per 100,000 parts of flock.

Black's method (*Analyst*, 1913, XXXVIII, 409). *Solutions required :* N/10 silver nitrate solution. N/10 potassium sulpho-cyanide solution.

The process. 40 grams of the flock are placed on a Buchner funnel fitted to a 58 oz. flask, and thoroughly wetted with distilled water until the mass can be pressed down as a thick felt ; the mass is then covered with water and allowed to stand ; after standing the water is passed through the flock by pressure and the washing continued until the filtrate reaches 2000 c.c. ; to 500 c.c. of the filtrate a few drops of perhydrol are added and the mixture is evaporated to small bulk ; 5 c.c. of concentrated nitric acid, and 20 c.c. of the N/10 silver nitrate solution, are then added ; the mixture is heated to 90° C., cooled, agitated and filtered ; the precipitate is washed, and the filtrate titrated with the N/10 potassium sulpho-cyanide solution, using a saturated solution of iron alum as an indicator, until a permanent red colour is obtained. Each c.c. of silver nitrate solution used represents 0·00355 gram of soluble chlorine.

URINE.

Urine has a yellow colour, if normal; in the presence of blood it has a brownish yellow smoky appearance and, in the presence of bile, a green colour; turbidity is sometimes due to the presence of urates, phosphates or mucus. It is essential that the amount of urine daily voided should be known, for the analysis of urine to be of any value. The complete analysis of urine is a matter of very great difficulty, but the most important constituents, urea, chlorides, sugar and albumen, can be determined with sufficient accuracy.

Qualitative examination. *Reaction.* Normal urine should be acid to litmus but it quickly becomes alkaline owing to the formation of ammonium carbonate by the fermentation of the urea.

Albumen. Make a few c.c. of the urine faintly acid with acetic acid and boil, albumen is precipitated, and appears as a faint cloud; proteoses, etc. may be precipitated but redissolve on heating.

Picric acid test. To a few c.c. of the urine add an equal quantity of picric acid solution (containing 10 grams of picric acid and 20 grams of citric acid in 1000 c.c.); in the presence of albumen an immediate precipitate is formed.

Detection of sugar. 5 c.c. of the urine (boiled and filtered in the presence of albumen) are mixed with 5 c.c. of mixed Fehling's solution and raised to boiling; in the presence of sugar a red precipitate of cuprous oxide is obtained. Or 5 c.c. of the urine may be mixed with 5 c.c. of saturated picric acid solution, and 2 c.c. of a 6 per cent. solution of potassium hydroxide added; the mixture is then heated to boiling, when, if the urine is, for clinical purposes, free from sugar a bright red colour appears in the transparent solution when held up to the light; but, in the presence of sugar, the colour becomes so intense as to cause the solution to be quite opaque.

Detection of bile. 5 c.c. of the urine are mixed cautiously with 5 c.c. of concentrated sulphuric acid and cooled; a small quantity of powdered cane sugar is added and the solution mixed and slightly warmed; in the presence of bile a reddish

violet colour is produced. Or some of the urine may be filtered through a white filter paper, and the paper allowed to dry, when dry a drop of yellow nitric acid is applied from the end of a glass rod to the centre of the paper; in the presence of bile, colour rings of violet, red, green, and yellow appear on the paper.

Detection of blood. A reddish smoky appearance in urine is characteristic of the presence of blood. To 5 c.c. of the urine add 5 c.c. of freshly prepared tincture of guaiacum (made from unoxidized resin), and 5 c.c. of an ethereal solution of hydrogen peroxide; in the presence of blood a deep blue colour is produced. Too much reliance must not be placed on this test as many other organic bodies will produce the colour.

Examination of the deposit. Some of the urine is allowed to stand in a conical glass and the deposit, which is generally simple in character, examined by the following tests.

1. Warm a little of the deposit in the urine, if it dissolves it consists entirely of urates.

2. If the deposit does not dissolve on warming, allow it to settle; pour off the urine and warm the deposit with acetic acid; if it dissolves it consists of phosphates.

3. If the deposit is insoluble in acetic acid, add a little hydrochloric acid and warm again; if it dissolves it is calcium oxalate.

4. If the deposit is still insoluble, it is probably uric acid; to a fresh portion of the deposit add a few drops of potassium hydroxide solution, uric acid will dissolve; or place a small quantity of the deposit in a small white dish, add a few drops of concentrated nitric acid and evaporate to dryness; cool and, when cold, add to the dry residue a drop of ammonia solution; in the presence of uric acid a purple colour is produced which turns to violet on the addition of a drop of potassium hydroxide solution.

The deposit should also be examined microscopically; this examination requires a large experience to be of any value, but the general characteristics of some of the deposits are as follows:

Uric acid. These crystals are flat plates, dumb-bell-shaped, stellate, needle-shaped or rosetted; if a drop of potassium

hydroxide solution is inserted under the cover slip, they dissolve.

Ammonium urate. It occurs in small tufts of needle-shaped crystals; these should be dissolved in a drop of water on the slide and a drop of acetic acid added; the mixture warmed slightly and allowed to cool, when the characteristic uric acid crystals will be obtained.

Calcium oxalate. The crystals are generally small and octahedral; if a drop of acetic acid is inserted under the cover glass they do not dissolve but will do so with a drop of hydrochloric acid.

Magnesium ammonium phosphate. The crystals appear in the form of stellate plates immediately the urine becomes, or is made, alkaline.

Cystin appears as flat hexagonal crystals which are soluble in ammonia.

Blood may be recognized by the circular red corpuscles which are sometimes angular in old or stale urine.

Pus corpuscles are light coloured and slightly larger than those of blood and appear in the deposit as a light coloured layer.

Quantitative Examination of Urine.

Determination of specific gravity. 10 c.c. of the urine are measured out into a weighed beaker and weighed; the weight of the urine multiplied by 100 gives the specific gravity; or it may be taken with a good hydrometer.

Example: 10 c.c. of a sample of urine weighed 10·249 grams; then the specific gravity of the sample was

$$10{\cdot}249 \times 100 = 1024{\cdot}9.$$

Estimation of the chlorides. 10 c.c. of the urine and 1 gram of pure powdered ammonium nitrate are evaporated to dryness and thoroughly charred; the char is extracted with hot water, filtered and well washed on the filter paper; the filtrate is acidified with dilute acetic acid to destroy the soluble carbonates; and the excess of acetic acid is neutralized with calcium carbonate; a drop or two of potassium chromate

solution are added, followed by N/10 silver nitrate from a burette until a permanent red colour is obtained. Each c.c. of N/10 silver nitrate used = 0·005845 gram of sodium chloride.

Example: 10 c.c. of urine required 11·7 c.c. of N/10 silver nitrate solution to produce a permanent red colour; then $11{\cdot}7 \times 0{\cdot}005845 = 0{\cdot}0683885 \times 100 = 6{\cdot}84$ parts of sodium chloride per 1000 parts of urine.

Estimation of urea. Russell and West (*Journ. Chem. Soc.* 1859, XII, 749) devised an exceedingly simple apparatus consisting of a decomposition tube 9 ins. long and about half an inch in diameter, with a constriction near the closed end, and the open end fitted into a pneumatic trough, supported on legs; and a measuring tube, holding about 40 c.c.

Solutions required: Solution of sodium hypobromite is obtained by dissolving 100 grams of sodium hydroxide in 250 c.c. of water; and, just before the experiment, mixing 25 c.c. of this solution with 2·5 c.c. of bromine.

The process. 5 c.c. of the urine are carefully placed in the decomposition tube and rinsed in with water, until the bulb is full; the constriction is then plugged with a glass rod; the hypobromite solution is now run into the tube and the measuring tube is filled with water and inverted in the trough; the glass rod is removed and the measuring tube instantly placed over the decomposition tube, the hypobromite reacts with the urea to form nitrogen, which escapes into the measuring tube; at the end of 15 minutes, the volume of gas in the measuring tube is noted, and this volume divided by 371 gives the number of grams of urea in the 5 c.c. of urine taken. This method obviates calculation for temperature and pressure, as Russell and West found that 0·1 gram of urea constantly gave 37·1 c.c. of nitrogen at ordinary temperatures and pressures.

Example: In an experiment 5 c.c. of urine evolved 27·3 c.c. of nitrogen, then $\frac{27{\cdot}3}{371} = 0{\cdot}0736 \times 200 = 14{\cdot}72$ grams of urea in 1000 parts of urine.

Estimation of sugar. *Solution required:* Pavy's solution. 60 c.c. of Fehling's copper solution (p. 87) and 60 c.c. of Fehling's alkaline solution are mixed with 300 c.c. of ammonia solution

(specific gravity 0·880) and 10 c.c. of a 10 per cent. solution of sodium hydroxide in a 1000 c.c. flask, and made up to the mark with water. Each c.c. of this solution corresponds to 0·0005 gram of sugar calculated as glucose.

The process. 100 c.c. of the Pavy solution are placed in a boiling flask, fitted with an outlet tube, to which is attached a piece of rubber tubing leading out of the window or into a fume cupboard to carry away the fumes of ammonia. 10 c.c. of the filtered urine are placed in a 200 c.c. flask, and made up to the mark with water; this diluted urine is cautiously delivered from a burette, which is attached to the boiling flask by means of a piece of rubber tubing, into the boiling Pavy solution until the solution is colourless; the volume of diluted urine required to decolourize the Pavy solution is noted, and this contains 0·05 gram of sugar calculated as glucose.

Example: 35 c.c. of diluted urine were required to decolourize 100 c.c. of Pavy's solution; then 35 c.c. of the diluted urine contain 0·05 gram of sugar, therefore the original urine contains $\frac{0{\cdot}05 \times 200 \times 100}{35} = 28{\cdot}57$ parts of sugar calculated as glucose per 1000 parts of urine.

Estimation of albumen. *Solution required:* Esbach's solution; 10 grams of picric acid, and 20 grams of citric acid, per 1000 c.c.

The process. Esbach's albumenometer is a specially graduated tube with two marks *U* and *R*; the urine is poured into the tube to the mark *U* followed by the Esbach's solution to the mark *R*; the tube is then closed by a rubber bung and the contents are gently mixed, but not shaken; the tube is now set aside for 24 hours and the height of the coagulum read off on the graduations, which represent parts of dry albumen per 1000 parts of urine. If the percentage of albumen is high, an equal volume of water should be added to the urine, and the result multiplied by two. If the urine is alkaline, it should be made acid with acetic acid. The albumen in a urine with less than 0·5 part per 1000 cannot be accurately estimated by this method.

Alternative process. A quantity of the urine is filtered; made faintly acid with acetic acid, and the specific gravity

taken ; the acidified urine is boiled and filtered and the specific gravity of the filtrate taken ; the specific gravity of the unboiled urine less the specific gravity of the boiled urine multiplied by four gives the number of grams of albumen in 1000 parts of urine.

Example : The specific gravity of an unboiled urine was 1029 ; and the specific gravity of the boiled urine was 1022 ; then $(1029 - 1022) \times 4 = 28$ parts of albumen per 1000 parts of urine.

Estimation of total phosphates. *Solutions required :* Standard uranium nitrate solution containing 35·5 grams of uranium nitrate in 1000 c.c. Each c.c. of this solution = 0·005 gram of phosphate (as P_2O_5).

Acid sodium acetate solution ; 100 grams of sodium acetate and 100 c.c. of glacial acetic acid in 1000 c.c.

Tincture of cochineal.

The process. 50 c.c. of urine and 5 c.c. of the acid sodium acetate solution are heated to 80° C. ; to the hot solution a few drops of the tincture of cochineal are added and the standard solution of uranium nitrate is run in from a burette until the red colour of the tincture of cochineal is changed to green. The number of c.c. of the uranium nitrate solution used multiplied by 0·005 multiplied by two gives the amount of phosphate (as P_2O_5) in 100 c.c. of the urine.

Estimation of phosphate combined with calcium and magnesium. 200 c.c. of the urine are made alkaline with ammonia and allowed to stand for 12 hours ; the mixture is then filtered and the precipitate washed with dilute nitric acid (1 in 3) ; the precipitate is then dissolved in a few c.c. of acetic acid ; 5 c.c. of the acid sodium acetate solution are added, and the whole made up to 50 c.c. ; the phosphate is then determined as described above. The total phosphate less the phosphate combined with calcium and magnesium gives the phosphate combined with the alkalies.

Estimation of sulphate. 100 c.c. of the urine are placed in a beaker ; 5 c.c. of concentrated hydrochloric acid are added and the mixture is boiled ; to the boiling solution, excess of barium chloride solution is added and the precipitate is allowed

to settle; the precipitate is then filtered off, washed with hot water, dried in a water oven, incinerated and weighed; the weight of barium sulphate obtained multiplied by 0·412 gives the weight of sulphate (as SO_4) in 100 c.c. of the urine.

Estimation of uric acid. *Solution required:* N/10 potassium permanganate solution containing 1·581 grams of potassium permanganate in 1000 c.c.

The process. To 100 c.c. of the urine add 25 grams of ammonium chloride; stir the mixture briskly until the ammonium chloride is dissolved; add 2 c.c. of strong ammonia solution and allow the solution to stand until the precipitate of ammonium urate has settled; filter and wash the precipitate with ammonium chloride solution; transfer the precipitate with boiling water (not more than 20 to 30 c.c.) to a beaker; add 1 c.c. of concentrated hydrochloric acid and heat the solution to boiling; allow the solution to stand until the uric acid has crystallized out (generally overnight); filter; measure the volume of the mother liquid; wash the precipitate with cold water; transfer it to a beaker and dissolve it in 1 c.c. of a 10 per cent. hot solution of sodium carbonate, and 100 c.c. of distilled water; to the solution add 20 c.c. of concentrated sulphuric acid, and titrate the mixture with the standard solution of potassium permanganate, with vigorous agitation, until a permanent pink colour is obtained. Each c.c. of the potassium permanganate solution used represents 0·00375 gram of uric acid; to the amount of uric acid obtained must be added 0·001 gram for each 15 c.c. of mother liquor.

Example: The uric acid obtained from 100 c.c. of a urine required 20·1 c.c. of the standard potassium permanganate solution to produce a permanent pink colour; and 20 c.c. of mother liquor were obtained; then

$$(20{\cdot}1 \times 0{\cdot}00375) + (0{\cdot}001 \times 20/15) = 0{\cdot}0767 \text{ gram}$$

of uric acid in 100 c.c. of the urine.

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In addition to the various references to periodicals, memoirs and reports in the preceding pages, the following list gives the titles of books which have been consulted from time to time during the laboratory experience of the authors of this book. The list does not profess to mention the names of all the volumes and memoirs which have been published dealing with the different subjects ; but probably it contains the most useful.

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ADDENDA

Table of atomic weights.

1913

International Atomic Weights.

		O = 16			O = 16
Aluminium	Al	27·1	Lithium	Li	6·94
Antimony	Sb	120·2	Magnesium	Mg	24·32
Arsenic	As	74·96	Manganese	Mn	54·93
Barium	Ba	137·37	Mercury	Hg	200·6
Bismuth	Bi	208·0	Nickel	Ni	58·68
Boron	B	11·0	Nitrogen	N	14·01
Bromine	Br	79·92	Oxygen	O	16·00
Cadmium	Cd	112·40	Phosphorus	P	31·04
Calcium	Ca	40·07	Platinum	Pt	195·2
Carbon	C	12·00	Potassium	K	39·10
Chlorine	Cl	35·46	Silicon	Si	28·3
Chromium	Cr	52·0	Silver	Ag	107·88
Cobalt	Co	58·97	Sodium	Na	23·00
Copper	Cu	63·57	Strontium	Sr	87·63
Fluorine	F	19·0	Sulphur	S	32·07
Gold	Au	197·2	Thallium	Tl	204·0
Hydrogen	H	1·008	Tin	Sn	119·0
Iodine	I	126·92	Titanium	Ti	48·1
Iron	Fe	55·84	Uranium	U	238·5
Lead	Pb	207·10	Zinc	Zn	65·37

Table containing the percentages of absolute alcohol by volume and weight obtained from a determination of the specific gravity at 15° C.

Specific gravity at 15° C.	Vols. per cent.	Weight per cent.	Specific gravity at 15° C.	Vols. per cent.	Weight per cent.
1·0000	0	0	·9362	49·00	41·55
·9999	0·05	0·05	·9343	50·00	42·50
·9998	0·15	0·10	·9324	51·00	43·45
·9991	0·55	0·45	·9303	52·00	44·40
·9985	1·00	0·80	·9283	53·00	45·35
·9970	2·00	1·60	·9263	54·00	46·25
·9956	3·00	2·40	·9242	55·00	47·25
·9942	4·00	3·20	·9221	56·00	48·20
·9929	5·00	3·95	·9199	57·00	49·20
·9915	6·00	4·80	·9178	58·00	50·20
·9902	7·00	5·65	·9157	59·00	51·15
·9890	8·00	6·40	·9134	60·00	52·20
·9878	9·00	7·25	·9111	61·00	53·20
·9866	10·00	8·05	·9089	62·00	54·15
·9854	11·00	8·90	·9066	63·00	55·15
·9843	12·05	9·75	·9043	64·00	56·20
·9832	13·00	10·50	·9020	65·00	57·20
·9821	14·00	11·35	·8996	66·00	58·25
·9811	15·00	12·15	·8971	67·00	59·30
·9800	16·00	13·00	·8949	68·00	60·30
·9790	17·00	13·80	·8923	69·00	61·40
·9780	18·00	14·65	·8899	70·00	62·50
·9770	19·00	15·45	·8874	71·00	63·50
·9760	20·00	16·30	·8848	72·00	64·65
·9750	21·00	17·10	·8824	73·00	65·65
·9739	22·05	17·95	·8797	74·00	66·75
·9730	23·05	18·80	·8772	75·00	67·90
·9719	24·00	19·60	·8744	76·00	69·00
·9709	25·00	20·45	·8719	77·00	70·10
·9698	26·00	21·30	·8692	78·00	71·25
·9688	27·00	22·10	·8664	79·00	72·40
·9677	28·05	23·00	·8638	80·00	73·50
·9666	29·00	23·80	·8610	81·00	74·70
·9654	30·00	24·70	·8580	82·00	75·85
·9643	31·00	25·05	·8553	83·00	77·00
·9630	32·05	26·45	·8524	84·00	78·25
·9619	33·00	27·20	·8493	85·00	79·45
·9605	34·00	28·10	·8465	86·00	80·65
·9592	35·00	28·95	·8433	87·00	81·90
·9578	36·00	29·85	·8402	88·00	83·10
·9564	37·00	30·70	·8369	89·00	84·45
·9549	38·00	31·60	·8337	90·00	85·65
·9533	39·05	32·50	·8304	91·00	87·00
·9519	40·00	33·35	·8269	92·00	88·35
·9503	41·00	34·25	·8237	93·00	89·60
·9487	42·00	35·15	·8199	94·00	91·00
·9470	43·00	36·05	·8160	95·00	92·45
·9453	44·00	36·95	·8123	96·00	93·80
·9436	45·00	37·85	·8081	97·00	95·30
·9417	46·00	38·80	·8036	98·00	96·85
·9406	47·00	39·70	·7988	99·00	98·40
·9381	48·00	40·60	·7938	100·00	100·00

Table showing the volumes of oxygen and nitrogen absorbed from the atmosphere by distilled water and by sea-water at 760 mm., and at various temperatures. The numbers are in c.c. per litre.

Temperature ° C.	Dittmar (*Challenger Reports*, Physics and Chemistry, vol. 1, p. 160). Sea-water		Distilled water		Roscoe and Lunt (*Jour. Chem. Soc.* 1889, p. 552). Distilled water	
	Oxygen	Nitrogen	Oxygen	Nitrogen	Oxygen	Nitrogen
− 5	9·42	17·85				
− 4	9·14	17·35				
− 3	8·89	16·37				
− 2	8·63	16·43				
− 1	8·40	16·00				
0	8·18	15·60	10·24	19·30		
+ 1	7·97	15·21	9·97	18·86		
2	7·77	14·85	9·71	18·35		
3	7·58	14·50	9·46	17·91		
4	7·39	14·18	9·22	17·50		
5	7·22	13·86	8·99	17·09	8·68	
6	7·05	13·56	8·78	16·72	8·49	
7	6·90	13·26	8·57	16·36	8·31	
8	6·74	12·99	8·37	16·02	8·13	
9	6·59	12·73	8·18	15·69	7·95	
10	6·45	12·47	8·00	15·37	7·77 (7·87)	(15·47)
11	6·31	12·23	7·82	15·07	7·6	
12	6·19	11·99	7·66	14·77	7·44	
13	6·06	11·77	7·50	14·49	7·28	
14	5·94	11·56	7·35	14·22	7·12	
15	5·83	11·34	7·20	13·96	6·96 (7·09)	(13·83)
16	5·72	11·14	7·06	13·71	6·82	
17	5·61	10·95	6·92	13·47	6·68	
18	5·51	10·76	6·78	13·25	6·54	
19	5·41	10·58	6·66	13·01	6·40	
20	5·31	10·41	6·53	12·80	6·28 (6·44)	(12·76)
21	5·22	10·24	6·41	12·60	6·16	
22	5·13	10·07	6·30	12·39	6·04	
23	5·04	9·92	6·18	12·20	5·94	
24	4·95	9·77	6·07	12·01	5·84	
25	4·87	9·62	5·97	11·83	5·76 (5·91)	(11·78)
26	4·79	9·48	5·87	11·65	5·68	
27	4·71	9·34	5·77	11·48		
28	4·64	9·20	5·68	11·31		
29	4·55	9·07	5·58	11·15		
30	4·50	8·94	5·495	10·955		
31	4·43	8·82	5·41	10·84		
32	4·36	8·70	5·32	10·69		
33	4·30	8·58	5·24	10·55		
34	4·23	8·47	5·16	10·41		
35	4·17	8·36	5·08	10·28		

Table containing some useful constants.

1 metre = 39·37 inches.
1 cubic centimetre = 0·061 cubic inch.
28·35 cubic centimetres = 1 fluid ounce.
1000 cubic centimetres = 1 litre.
1 litre = 35·3 ounces = 1·765 pints.
1 gram = 15·432 grains.
1 ounce = 28·35 grams.
16 ounces = 453·6 grams.
1 gallon of water = 4·536 litres = 10 pounds.
1 litre of hydrogen at 0° C. and 760 mm. weighs 0·0896 gram.
1 c.c. of distilled water at 4° C. and 760 mm. weighs 1 gram.
To convert pints into litres, $\times 0{\cdot}568$.
To convert grains to grams, $\times 0{\cdot}0648$.
To convert Centigrade scale to Fahrenheit scale, $\times 9 \div 5$, and add 32.
To convert Fahrenheit scale to Centigrade scale, subtract 32, $\div 9 \times 5$.

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